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DESIGN AND DEVELOPMENT OF POLYPHENYLENE OXIDE FOAM AS A REUSABLE INTERNAL INSULATION FOR LH₂ TANKS

FINAL REPORT

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ABSTRACT

A 48 month program was conducted to evaluate and develop polyphenylene oxide (PPO) foam as an internal cryogenic gas layer insulation for LH_2 tanks. An evaluation was made of new PPO foam compositions produced by TNO in Delft, Holland. This evaluation resulted in efforts of the vendor to continue to improve the quality of the foam. The vendor varied the blowing agent, the nucleating agent, and the millsheet manufacturing methods, while GD/Convair performed detailed qualitative and quantitative evaluation of the panels produced. The work included preparation of a material specification and fabrication process procedures. The properties of mechanical strength, modulus of elasticity, density and thermal conductivity were measured and related to foam quality. Properties unique to PPO foam as a gas layer insulation; density gradient parallel to the fiber direction and gas flow conductance in both directions were correlated with foam quality.

FOREWORD

This report was prepared by Convair division of General Dynamics Corporation. San Diego, California for NASA, Marshall Space Flight Center. This is a final contract report which presents the results of Contract NAS8-27566, "Design and Development of Polyphenylene Oxide Foam as a Reusable Internal Insulation for LH₂ Tanks." The work was performed during the period July 1971 to June 1975.

This contract was administered under the technical direction of Dr. James Stuckey and Mr. L. M. Thompson, S&E-ASTN-MNM, Astronautics Laboratory of NASA-MSFC.

The General Dynamics Convair personnel who made major contributions to the program. Mr. R. E. Tatro is Program Manager; F. O. Bennett, material characterization, inspection and analysis; H. G. Prittian, cryogenic and environmental testing; M. Maximovich, adhesives and bonding development; P. Merz, rigidization and chemical processes; R. L. Otwell, repair and joint design; C. Snyder, fabrication and repair tooling; G. B. Yates, insulation design, specification and thermal analysis.

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SUMMARY

Convair Division of General Dynamics Corporation has conducted a 48 month program to evaluate and develop polyphenylene oxide (PPO) foam as an internal cryogenic gas layer insulation under contract NAS8-27566, "Design and Development of Polyphenylene Oxide Foam as a Reusable Internal Insulation for LH₂ Tanks". The work included preparation of a material specification and fabrication process procedures. Mechanical strength, modulus of elasticity, density and thermal conductivity were measured and related to foam quality. Density gradient parallel to the fiber direction and gas flow conductance in both directions were also correlated with foam quality. These are properties unique to PPO foam as a gas layer insulation.

New PPO foam compositions produced by TNO in Delft, Holland were evaluated by Convair. The vendor continued to improve the quality of the foam. This resulted finally in the selection of an optimum composition. The vendor varied the blowing agent, the nucleating agent, and the millsheet manufacturing methods, while GD/Convair performed detailed qualitative and quantitative evaluation of the foam panels produced. Combinations of dichloroethane (DCE), trichloroethane (CNU), and the petroleum ether (SEB) were used as blowing agents with the addition of vermiculite (VER) or Genitron (GEN) as nucleating agents. Material used to determine thermal conductivity was subsequently subjected to pressure drop (permeability) and density gradient evaluations. A preferred composition was selected in 1972; the blowing agent used was a mixture of dichloroethane (DCE) and trichloroethane (CNU), and the nucleating agent was Genitron AC/2 azodicarbonamide added in the ratio of two parts per hundred parts of resin. The panels were foamed from rolled millsheets in an open press. This combination of blowing and nucleating agents was found to result in panels having the most uniform structure and lowest thermal conductivity. The rolled millsheet manufacturing method was chosen because extrusion or injection molding methods had not been refined to produce panels of consistent uniformity.

A material specification was prepared in sufficient detail to assure the production and delivery of foam which will provide a high quality long life insulation. All the material classifications which have been produced to date are included; densities from 30 kg/m³ (1.87 lb/ft³) to 50 kg/m³ (3.12 lbs/ft³) and thicknesses from 30 mm (1.18 in) to 185 mm (7.28 in.). Non-destructive test (NDT) are specified for all material and destructive quality control tests on statistical samples. The most effective NDT is X-ray to locate voids and other undesirable density gradients.

PPO foam has the anisotropic cellular configuration of honey-comb. It has a very low modulus of elasticity perpendicular to the fiber direction even at cryogenic temperature. As a gas layer insulation, it has no sealed inner surface. Butt joints are compressed as panels are installed thus do not require bonding. These characteristics combine

to make material relatively easy to install. While the repair of any bonded plastic material is a challenge, the only PPO foam bond to repair is at the tank surface. There are no joints or sealed membranes. A panel was repaired twice in the same place and tested six times over a period of about one year with a maximum degradation of only twenty percent.

The thermal conductivity of the foam when used as a gas layer insulation is, as a minimum, that of gaseous hydrogen (GH_2). Above a mean temperature of 153 K (275R), the best foam panels had a thermal conductivity that were only 10 percent higher than GH_2 . At very low temperatures, 56K (100R), the thermal conductivity was only 40 percent above the thermal conductivity of GH_2 .

The foam was thermal cycled and soaked from 21K (37R) to 450K (810R) in various gaseous environments. In a vacuum or non-oxidizing gas environment, the foam successfully withstood the high temperature environment and cycling. About two percent shrinkage occurs at 422K (760R). Therefore the foam should be pre-aged in an inert environment to the maximum predicted use temperature. The foam is expanded anisotropically from a flat thin sheet in a heated platen press to the desired thickness. Since the plates are in contact with both surfaces, the forces involved in the expansion cause the panels to stretch to a reduced density in the center. The center density reduction was correlated with gas flow conductance and thermal conductivity. A reward to say that a 10% reduction was not detrimental to thermal conductivity.

The foam cells consist of very thin membranes. When cut or impacted they tend to break and leave debris. This is undesirable for an internal propellant tank insulation. A radiant heating procedure was developed to harden the cell edges. This gives an impact resistant surface.

Lonza, Ltd., Basel, Switzerland, the PPO foam patent license holder, continued work through 1974 on processes to put PPO foam into automated production. However lack of a firm quantity order restrained the work to component development and paper production line studies. TNO, the original foam producer, has continued to produce the foam from rolled mill resin sheets. They have added edge closures to their foaming press and tightened the process quality control. In 1975, TNO produced foam which meets the GD/Convair specification for density gradients and had the lowest thermal conductivity in liquid hydrogen of any foam tested during this program. The resin was mixed with DCE/CNU blowing agent, 2 pph VER nucleating agent and blown from a single mill sheet.

SECTION 1

INTRODUCTION

The objective of this program was to evaluate polyphenylene oxide (PPO) foam as an internal insulation for LH₂ tanks. The system must withstand the Space Shuttle launch, earth orbit and reentry mission cycle, be capable of withstanding short-term exposure to a 450K (350F) environment, and be reusable for up to 100 flights with minimum refurbishment.

PPO foam is a unique anisotropic material with a cellular structure similar to honeycomb. When the material is used as an internal insulation, the combination of heat flux and surface tension limits liquid entry into the cells, thus forming an insulating gas layer. PPO foam was first demonstrated to be a feasible internal gas layer insulating material for liquid hydrogen in 1969 (Ref. 1). Its simplicity, in terms of handling and fabrication, and its unique properties led to further development of the material for liquid hydrogen use. Mechanical property, thermal conductivity, and small-scale forming tests were performed on the material and comparisons made with competitive liquid hydrogen tank insulations (Ref. 2). This work was prompted primarily by a search for an insulation that could withstand multiple reuses on cryogenic-fueled launch vehicles such as Space Shuttle. An internal gas layer insulation is desirable because it is less subject to handling damage, a "warm" bond line minimizes cyclic thermal stresses, and there is no pressure load on the insulation. A 1,000-gallon tank was built specifically to evaluate the thermal and mechanical integrity of PPO foam insulation. The insulation was successfully subjected to 100 tanking, pressurization, detanking, and external heating cycles (Ref. 3). Several small liquid hydrogen test tanks were designed and built to verify the mechanical integrity of various insulation composites. One set of interchangeable heads, internally insulated with PPO foam, was built for these tanks. Large PPO foam panels were fabricated to demonstrate low-cost fabrication and handling ease (Ref. 4).

A PPO foam materials specification was prepared and process procedures for application of a complete PPO foam internal insulation system to a liquid hydrogen tank were written.

X-ray and photographic evaluation are the most convenient method of inspecting incoming panels of PPO foam. Internal voids and areas of large density gradients are easily recognizable. The procedures and techniques for making X-ray exposures and prints of the exposures were standardized to permit an accurate assessment of the material quality and to allow use of X-ray inspection to predict material thermal and structural characteristics.

Samples of PPO foam were subjected to long-term exposure at elevated temperatures to determine weight and dimensional stability, and to identify outgassing products. Other samples were subjected to 100 temperature cycles simulating the shuttle mission thermal environment to determine the effects on weight and dimensional stability.

Various PPO foam configurations, densities, and thicknesses were evaluated for mechanical strength, thermal performance, and internal cell structure. Both bonded and unbonded samples were thermally cycled to evaluate the effects on strength, porosity, surface hardness, and bond integrity. Lateral and longitudinal density gradients were measured. Foam permeability and porosity were investigated using room temperature gases, and the thermal conductivity in liquid hydrogen was measured using a guarded flat-plate calorimeter. Results were correlated to develop a procedure for predicting thermal performance without performing expensive thermal tests.

Existing information on adhesives was reviewed and candidates selected for screening. Lap shear tests were performed and bonded foam samples subjected to a comprehensive evaluation program to select an adhesive that meets shuttle requirements. Tension, shear, and peel mechanical tests, as well as thermal shock and cycling tests, were performed on PPO foam bonded samples.

When individual PPO foam panels are installed in a tank joints result. Potential joining techniques were evaluated for structural performance and thermal performance. Joints were subjected to thermal testing using a guarded flat plate thermal conductivity apparatus. Previous close-out material investigations were reviewed and data on other candidate materials assembled. Methods for cutting, fitting, and bonding the materials were investigated. Methods were developed for applying PPO foam panels to both flat and curved surfaces. Improvements made in cutting, forming, and bonding techniques and effects of each on system structural and thermal performance were evaluated. A method for foam surface rigidizing was developed as well as techniques for tank surface preparation and bonding. The effect of various primers on bond integrity at temperatures from 21K to 450K (-423F to 350F) was determined. Techniques to be used for repairing insulation inside a tank were investigated. Methods for removal of damaged foam areas and the adhesive were investigated. Installation procedures for new foam were developed. Repaired areas were evaluated for changes in thermal performance.

The PPO foam vendor continued a program of improving panel quality and consistency. More promising compositions were selected for evaluation based on investigations by both the vendor and GD/Convair. These compositions were subjected to a screening program consisting of a determination of the effect of long term exposure to 450K (350F), the longitudinal and lateral permeability and density variations, the thermal conductivity, and the tensile and compressive strengths.

Although the English system of units (ft. lb. sec) has been used for all measurements and calculations, in this report the S.I. (m, kg, sec) system of units is shown as the primary system with English units following in parentheses.

SECTION 2

SPECIFICATIONS AND PROCESSES

This section contains the materials specification and fabrication procedures for a PPO foam internal cryogenic insulation system. The system operating environments in particular the thermal environment of atmospheric entry, tend to be severe compared with those experienced by existing insulation systems, and the requirements for the system to be reusable for up to 100 flights with minimum refurbishment demands for the use of materials of the highest quality and the use of properly qualified insulation application procedures.

Fabrication processes necessary for installation of the foam in an internally insulated tank are detailed. These include cutting, foaming, and bonding. A detailed description of the recommended repair procedures is given in Section 9.

2.1 MATERIAL SPECIFICATION

A preliminary specification for the procurement and qualification of PPO foam has been prepared, Appendix C. This specification is intended to provide a means of procuring high quality foam for use as an internal insulation in a liquid hydrogen tank. The specification provides for the measurement and control of the physical and mechanical properties as well as the ingredients used in the raw materials prior to foaming. Sampling, qualification and acceptance tests, records and reporting, handling and shipping are specified. The specification is preliminary at this time because a completely qualified vendor, capable of large scale production within the limits of the specification has not been identified. The material is currently produced only by the Plastics and Rubber Institute, TNO, Delft, Holland using a batch process which has limited built-in quality control.

2.2 FABRICATION PROCESSES.

Procedures for handling and bonding PPO foam panels have been developed. The procedures and processes currently in use are described herein.

2.2.1 PPO FOAM CUTTING

Horizontal Cuts (perpendicular to cells)

1. The surface is scored with a blade in a checkerboard pattern spaced 2.5 cm (1.0 in) apart in both the width and length directions. The cut should be made to a constant depth of 1.5 mm (0.060 in). This relieves the stresses and eliminates distortion in the panel during cutting.

2. Load the panel uniformly to approximately 0.86 kN/m^2 (0.125 psi).
3. There are two principle methods of making horizontal cuts (perpendicular to the cells) in the foam: using a horizontal band saw or a circular rotary knife. These techniques are :
 - a. Horizontal Band Saw - The blade consists of 1.57 teeth per cm (4 teeth per inch) with no set in the teeth. The blade is run in reverse to minimize material tearing. Material is fed past the blade at a rate of 25.4 cm/min (10 in/min). After cutting, the material is vacuumed to remove debris.
 - b. Rotary Knife - A standard rotary knife, 5 cm (2 in) in diameter, is used at a rotation speed of 1800 rpm. The panel is fed past the knife at a speed of 76 cm/min (30 in/min) such that the cut is made into the material. The small amount of debris created is removed by vacuuming the surface either during or after cutting.

Vertical Cuts (parallel to cells)

Vertical cuts are made with the bandsaw. After cutting, the edges are sanded to remove loose material.

2.2.2 PPO FOAM FORMING

Tooling

1. All tools should be made of material which will withstand temperatures up to 450K (350F).
2. Mold should be of female configuration.
3. Edge supports to be made from a minimum of 6.35 mm (0.25 in) thick angle and formed to mold contour.
4. Over-press to be a minimum of three-ply fiberglass high temperature layup.

Preparation

1. Determine that mold face is free of contaminants, nicks, and distortions.
 - a. Remove contaminants from mold using sandpaper or wastecloth moistened with methyl ethyl ketone (MEK) or acetone.
 - b. Allow mold to air dry as necessary.

2. Cut the PPO foam panel in the desired size with a clean sharp hand saw blade, leaving an excess trim edge of approximately 1.26 cm (0.5 in) around perimeter of part.
3. Position the PPO foam panel on the mold.
 - a. Position edge supports around the perimeter of PPO foam part.
 - b. Position over-press over top of PPO foam part.

Forming Procedure

1. Apply the vacuum bag per GD/Convair manufacturing specification 85.24.2.
2. Apply vacuum slowly, removing as many bag wrinkles as possible.
3. Apply a vacuum of approximately 27.9 mm (11 inches) of mercury.
4. Form PPO foam part under a vacuum.
 - a. The oven temperature shall be $422\text{K} \pm 6\text{K}$ ($300\text{F} \pm 10\text{F}$) with a form time of 90 ± 10 min.
 - b. Let PPO foam part cool to ambient temperature under a vacuum.
 - c. Remove the formed panel from the mold.

2.2.3 PPO FOAM BONDING.

Preparation

1. All PPO surfaces requiring adhesive shall be free of loose particles. Thoroughly clean and dry at time of adhesive application.
2. All aluminum surfaces requiring adhesive shall be free of burrs; thoroughly clean and dry at time of adhesive application.
3. PPO foam and aluminum mating surfaces requiring adhesive shall be capable of continuous contact over total bond mating surfaces when pressed together clean, and dry at time of adhesive application.

4. PPO surface not requiring adhesive shall be protected from contacting the adhesive with masking tape or equivalent covering.
5. Cleaning materials and processes shall have no harmful effect on the part materials and shall produce surfaces that are free of oil, grease, dirt, moisture, and other foreign matter.
6. Aluminum surfaces shall be solvent cleaned and chromic acid etched (Forest Products Laboratory or Pasa Jet 105 etchant).

Bonding Procedure

1. Prepare adhesive in accordance with material specifications.
2. Any application method may be used providing it will insure a continuous and adherent film which is free from air entrapment and other visual imperfections. Both mating surfaces shall have a coating of adhesive material to a wet film thickness of 7.5 to 25 μm (0.003 to 0.010 inch). The specified film thickness shall apply to each surface coat.
3. Assemble the PPO foam panels after coating with adhesive by pressing assembly together under a load of 3.45 kN/m^2 (0.5 psia) to insure close contact of the bonded surfaces.
4. Remove excess adhesive material from aluminum boundary of the mating surfaces with clean dry cheesecloth.
5. Edge supports with capability to compress butt joints on PPO foam up to four percent uniformly are required.
6. Cure the bonded assembly under pressure of 10.3 to 17.2 kN/m^2 (1.5 to 2.5 psi). If vacuum bagging method is used, edge supports are required to protect edge of PPO foam.

SECTION 3

PPO FOAM INSPECTION TECHNIQUES

To achieve a high level of thermal performance and reliability necessary for use as a cryogenic insulation material, open-celled foam must be free of large voids and high-density areas and be of consistent high quality. To verify that manufactured PPO foam panels are of sufficiently high quality for use in an internal insulation system, methods of inspecting the incoming panels have been devised. The various techniques for panel inspection may be classified as either non-destructive or destructive. The non-destructive techniques include:

- a. radiography, X-raying the panels prior to cover paper removal.
- b. visual inspection for surface irregularities after paper removal.
- c. visual inspection on a light table for density variations.
- d. magnifying and measuring surface cell characteristics.
- e. calculating density of full size panels.

Destructive test techniques include:

- a. sectioning the panels for internal density variation calculations.
- b. determining porosity by measuring the pressure drop through the material.
- c. measuring mechanical properties by performing tensile, compression, shear, and peel tests.
- d. measuring thermal properties by performing calorimeter and thermal cycling tests.

All of these techniques are used during the process of screening the various PPO foam configurations, and, during a production run, most or all of these would be employed periodically by the vendor to verify conformance with material standards.

The panels are manufactured with brown Kraft-type paper on the two surfaces contacting the press to allow for subsequent release from the press plates. The paper then serves to protect the panel surfaces during transit and to prohibit foreign matter from entering the cells. Prior to removal of the cover paper all of the

incoming panels are logged in, visually inspected for damage or voids at the edges, and X-rayed. The X-ray tube used is a Norelco MG 50 with a beryllium window and a 1.5 mm focal spot. The tube is operated at its lowest power setting, 10 kv at 15 ma, because of the very small mass of material in the low density foam. The film used is 35.6×43.2 cm (14×17 in) Eastman Kodak Type T medium grain or its equivalent. The tube is positioned a distance 102 cm (40 in.) from the film for exposure to minimize parallax distortion. For a given distance from the tube to the film the distortion or image offset at any point on the upper surface of the foam is dependent on the lateral distance from the vertical and the panel thickness. This is illustrated in Figure 3-1. At the maximum lateral distance from the vertical, 27.9 cm (11 in.), corresponding to one-half the diagonal length of the film, the image offset of the upper edge of a 75 mm (3 in.) thick panel is 2.26 cm (0.89 in.). Thus it is more difficult to determine the location or nature of irregularities near the edge of the X-ray.

Current foam panels are approximately 61×76 cm (24×30 in.) in size. Four 35.6×43.2 (14×17 in.) X-ray films are used to cover a panel surface (Figure 3-2). The panels are marked with an "x" at the center and four "T's" on a 25.4 cm (10 in.) radius as shown. The corners are marked with "UR" for "upper right," etc., as well as the panel and X-ray log numbers. The films are positioned underneath the quadrants of the panel such that each will contain the center "x" and two of the "T's" resulting in a 5 cm (2 in.) overlap with adjacent films. The X-ray tube is centered over the films which are individually exposed. The developed films can then be overlapped on a large light table to give a total picture of the internal structure of the panel.

This inspection procedure works well for the determination of density variations in any one exposure. However, different exposures do not provide an accurate indication of the relative bulk density. The X-ray machine is being operated at the minimum practical energy (voltage) level. This results in a limited controllable range. The small mass of foam being exposed still allows a relatively large energy transfer to the film. The inclusion of an X-ray standard in each exposure would permit a quantitative determination of the relative level of film exposure and relative bulk densities, although this could be a time consuming and expensive operation.

Samples of PPO foam were inspected using neutron radiographic techniques at the Convair Division in Fort Worth, Texas. Neutrons from a 2.8 mg ^{252}Cf Californium source were reduced to thermal energy in water. These thermal neutrons were collimated and passed through the PPO foam and onto the 4×6 in conversion screen. The activated screen exposed the adjacent film to 70 kev electrons. Using several film types and exposure times it was not possible to achieve acceptable defect identification.

Other non-destructive tests performed after the cover paper has been removed include light table inspection, surface magnification and photography, and density measurements.

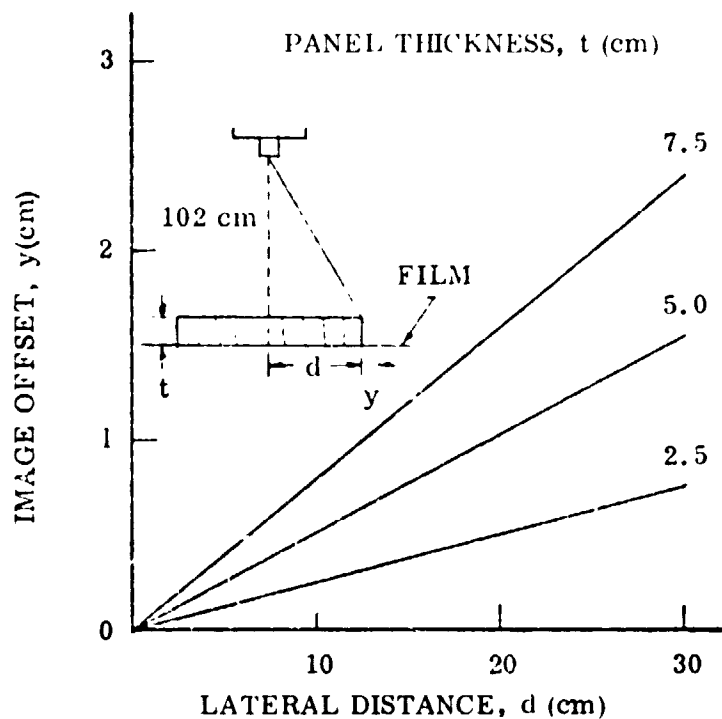


Figure 3-1. X-ray Image Offset Due to Parallax Effect

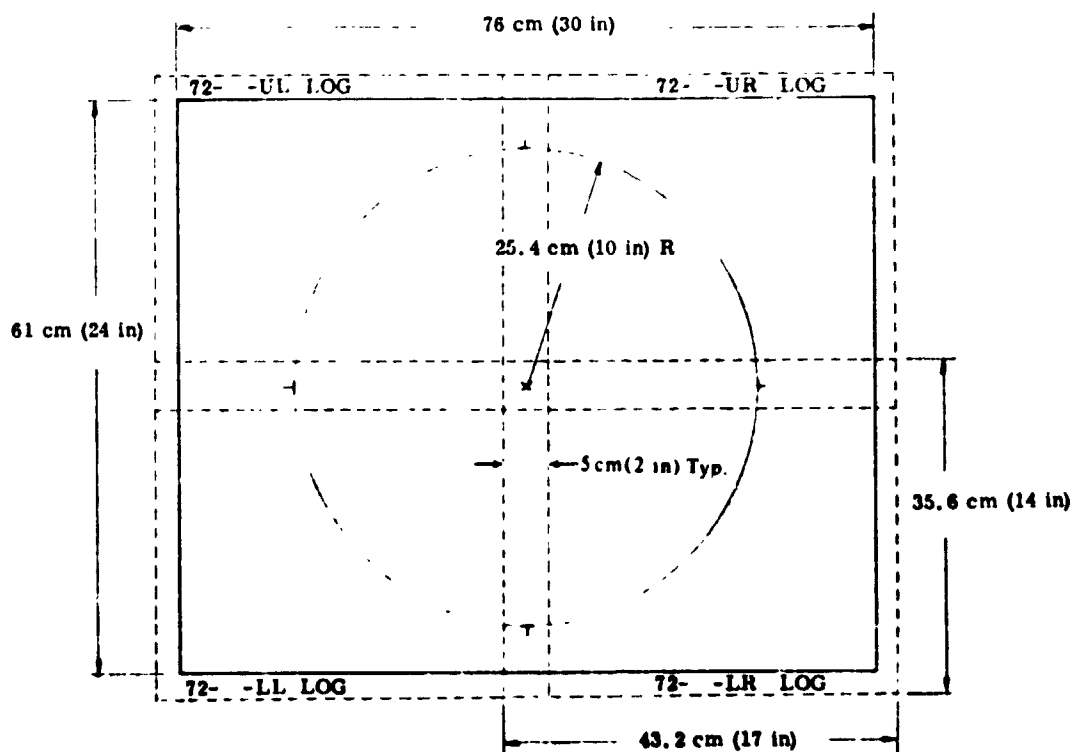


Figure 3-2. Location of X-ray Films to Cover Panel

117390B



Figure 3-3. Light Table Photograph of PPO
Foam Specimen

specimens for density variation, porosity, mechanical strength, and thermal conductivity determination. These techniques are described under the appropriate section headings in this report.

An example of light table photograph of a specimen used for thermal conductivity testing is shown in Figure 3-3. Differences in light transmittal can be caused by the degree of coarseness of cell structure or the number of closed cells as well as density variations. No definitive use of the light table inspection has been made. Details of the cell structure can be inspected by microphotographs taken of the surface and of cross sections through the material. Cell sizes (diameter) and configuration and the relative amount of debris can be determined by microphotograph. Finally, the calculation of bulk density of the trimmed panel is used to verify that the density is as specified.

The destructive test techniques involve sectioning the trimmed panel into

SECTION 4

MATERIAL PROPERTIES

This section presents the results of PPO foam panel density and density gradient measurements and mechanical strength tests.

4.1 DENSITY GRADIENTS

Due to the manner in which the foam is blown, the material in the middle of the panel (measured parallel to the cell orientation) is less dense than that near the surface and exhibits higher lateral permeability, i.e., lower resistance to the movement of gas in the direction perpendicular to the cell orientation. This reduced density and higher permeability results in a reduction of the mechanical strength of the foam and increased thermal conductivity due to instability of the gas layer caused by lateral gas movement. Ideally there should be no density gradients in a panel and the lateral permeability should be constant across the cross section. Then the bulk density of the panel could be adjusted such that the lateral permeability, which would then be proportional to the density, is maintained below a maximum allowable level.

Panels are first trimmed to the class I condition with the paper removed. The panels are then weighed, measured, and the nominal density computed. Each panel is sliced into three sheets corresponding to the upper, middle, and lower thirds, Figure 4-1. These sheets are weighed and measured and the densities calculated. These data provided a measurement of the average longitudinal density variation of the whole panel. Finally, each sheet is cut into sub panels each of which are weighed and measured. The densities of these pieces are then compared with each other and with the nominal panel density.

4.1.1 DEVELOPMENT MATERIAL. The standard blowing agent for PPO foam panels had been dichloroethane (DCE). In April 1971, a panel, 71-11, was received which utilized a 3:1 parts by volume mixture of Chlorothene Nu (CNU) (1,1,1 trichloroethane) and dichloroethane (DCE) as the blowing agent. This panel, $43 \times 33 \times 5$ cm ($17 \times 13 \times 2$ in.) with nominal density of 33 kg/m^3 (2.06 pcf), has been tested to determine the extent of both longitudinal (parallel to the fiber orientation) and lateral density variations. The results of the investigation are summarized below.

Measured nominal panel density	33.0 kg/m^3
Maximum longitudinal density variation from nominal	-6 percent
Maximum lateral density variation from nominal	3 percent
Maximum density variation of any piece from nominal	-11 percent

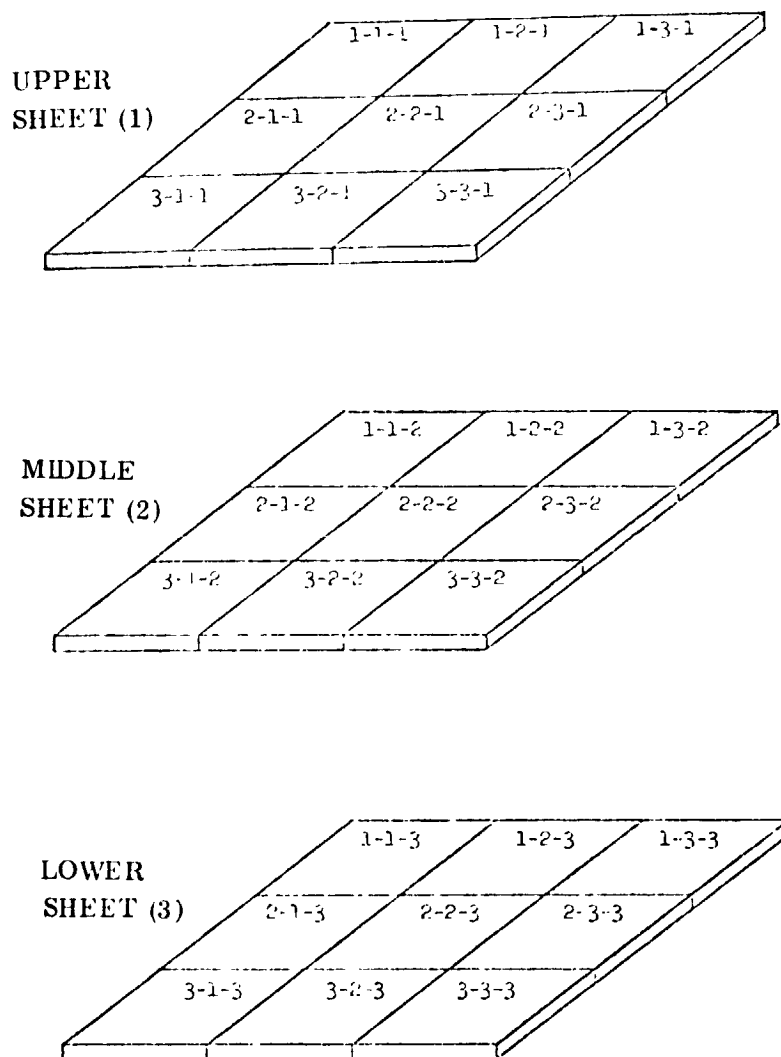


Figure 4-1. Identification of Cut Specimens (Sub Panels)

changes were made to correct the problem. The existence of the flecks had no apparent detrimental effect on the thermal or mechanical properties of the foam.

Two additional panel blown with DCE/CNU, panels 71-12 and -14 were evaluated. The as-received densities of these panels were 30.4 and 28.8 kg/m^3 (1.9 and 1.8 pcf). Both panels were cut and labeled in accordance with Figure 4-1. The surfaces of Panel 71-14 were left in the as received condition whereas approximately 2.54 mm (0.1 inch) was removed with a bandsaw from both surfaces of Panel 71-12. Panel 71-12 had a nominal density of 30.1 kg/m^3 (1.88 pcf) and Panel 71-14 had a nominal density of 28.8 kg/m^3 (1.8 pcf). The densities of the individual pieces were computed along with the percent variation from the nominal and are illustrated in Figures 4-5 and 4-6. The middle sheet of the panels had a lower density than the surface. The mean density of the middle sheet, Panel 71-14, was 24.5 kg/m^3 (1.53 pcf)

The density variations from the nominal value, 33.0 kg/m^3 (2.06 pcf), are shown in Figure 4-2. Note that the variations for the middle sheets are generally higher. The largest single variation from nominal is -11 percent. By combining the pieces as shown in Figure 4-3 and averaging the densities of the combined pieces, the maximum lateral variation from the nominal density was found to be 3 percent.

After the 27 pieces had been cut from the panel, it was noticed that small flecks of solid material occurred throughout the foam. These flecks, shown in Figure 4-4, are apparently pockets of resin that failed to expand during the blowing process possibly due to incomplete component mixing. This information was transmitted to the vendor where

UPPER
SHEET

-2	1	3
-1	1	4
1	2	3

NOMINAL DENSITY (ρ_N)
= 33.0 kg/m³ (2.06 pcf)

VARIATION

$$= \frac{\rho - \rho_N}{\rho_N} \times 100\%$$

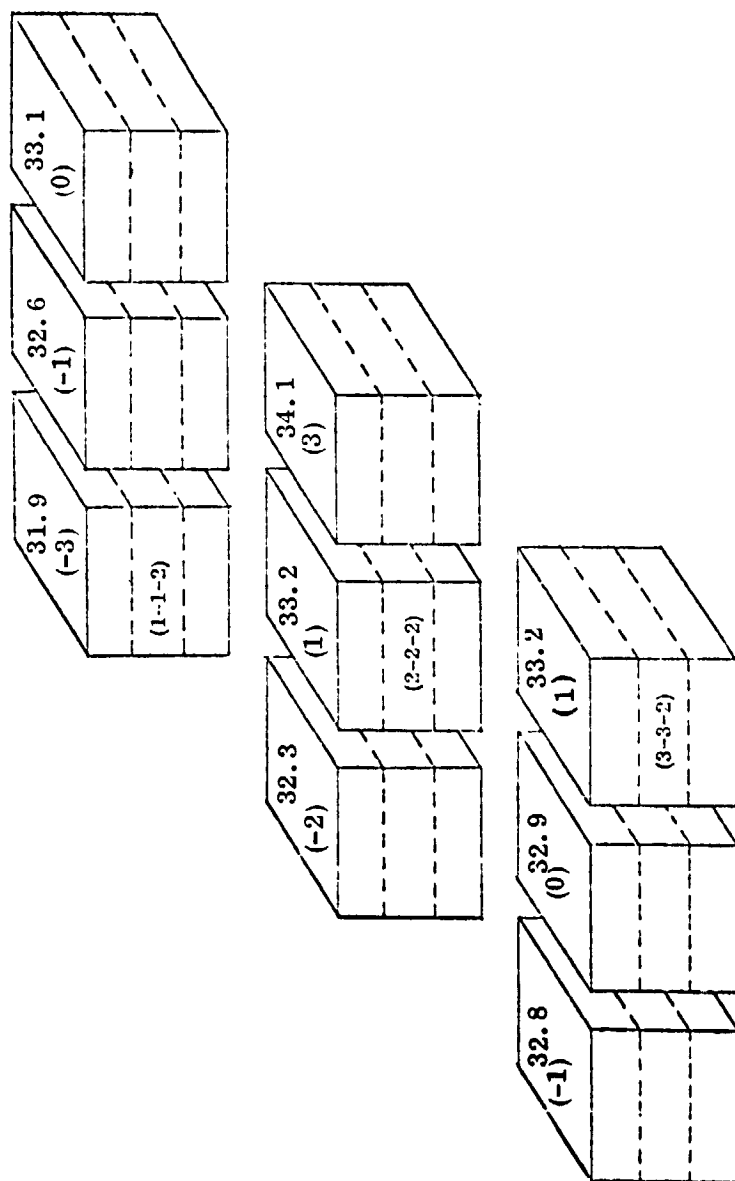
MIDDLE
SHEET

-11	-8	-8
-10	-5	-4
-8	-8	-5

LOWER
SHEET

2	4	6
5	6	10
6	5	7

Figure 4-2. Density Variations from Nominal, Panel 71-11



DENSITY IN KG/M³
 VARIATION IN PERCENT FROM NOMINAL (33.0 KG/M³)

Figure 4-3. Average Density of Three-Piece Stacks and Percent Variation From Nominal

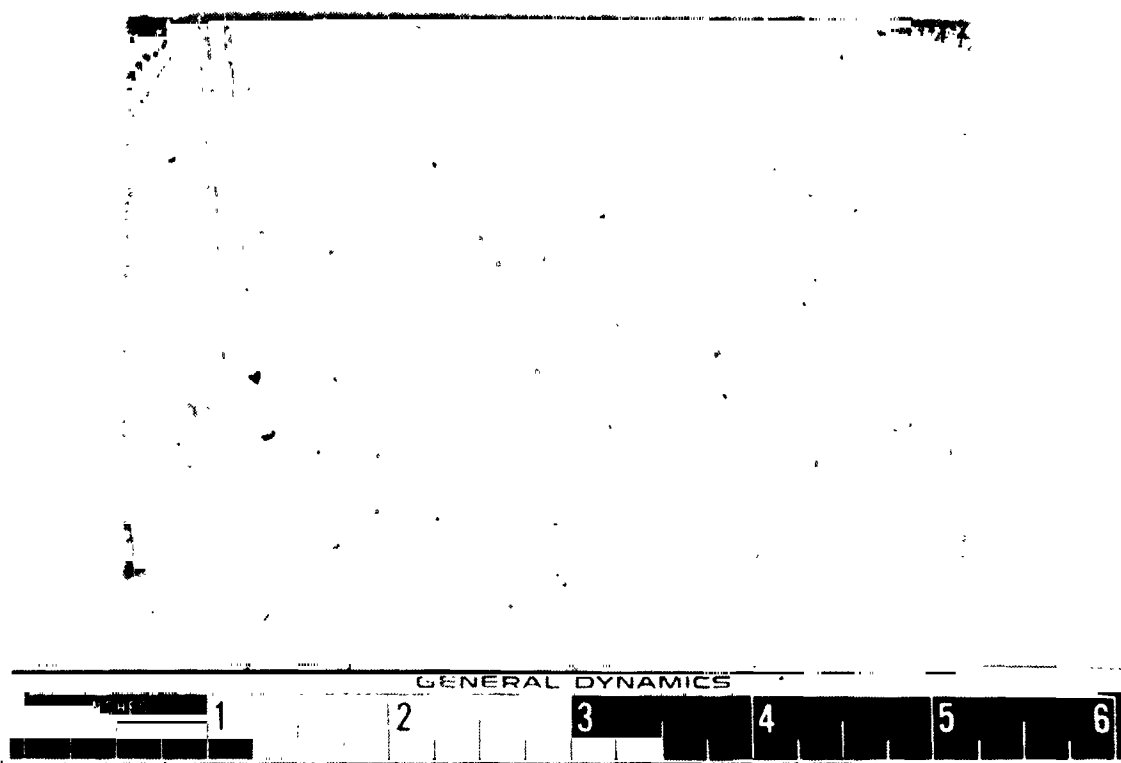


Figure 4-4. Solid Inclusion in the Foam Matrix

ORIGINAL PAGE IS
OF POOR QUALITY

PANEL 71-12

Density in pcf
(Variation)

UPPER SHEET
(1)

1.988 (+6)	2.050 (+11)	1.998 (+6)
2.027 (+8)	2.140 (+14)	1.985 (+5)
1.933 (+3)	2.042 (+9)	1.970 (+5)

Mean $\rho = 2.018$ pcf (+7)

Nominal Density = 1.882 pcf

$$\text{Variation} = \frac{\rho - \rho_N}{\rho_N} \times 100\%$$

MIDDLE SHEET
(2)

1.683 (-11)	1.765 (-6)	1.683 (-11)
1.673 (-11)	1.754 (-7)	1.665 (-12)
1.624 (-14)	1.698 (-10)	1.647 (-12)

Mean $\rho = 1.688$ pcf (-10)

LOWER SHEET
(3)

2.097 (+11)	2.159 (+15)	2.090 (+11)
2.166 (+15)	2.230 (+18)	2.081 (+11)
2.062 (+10)	2.100 (+12)	2.009 (+7)

Mean $\rho = 2.110$ pcf (+12)

Figure 4-5. Densities of Individual Pieces and Percent Variation From Nominal, Panel 71-12

PANEL 71-14

Density in pcf
(Variation)

UPPER SHEET
(1)

1.91 (+6)	1.89 (+5)	1.97 (+9)
1.89 (+5)	1.99 (+11)	2.07 (+15)
1.81 (+1)	2.0 (+11)	2.06 (+14)

Mean $\rho = 1.954$ pcf (+9)

Nominal Density = 1.8 pcf

Variation: $\frac{\rho - \rho_N}{\rho_N} \times 100\%$

MIDDLE SHEET
(2)

1.45 (-19)	1.41 (-22)	1.47 (-18)
1.58 (-12)	1.58 (-12)	1.65 (-8)
1.45 (-19)	1.54 (-14)	1.63 (-9)

Mean $\rho = 1.53$ pcf (-15)

LOWER SHEET
(3)

1.91 (+9)	2.08 (+16)	2.0 (+11)
2.02 (+12)	2.18 (+21)	2.14 (+19)
1.97 (+9)	2.18 (+21)	2.12 (+18)

Mean $\rho = 2.07$ pcf (+15)

Figure 4-6. Densities of Individual Specimens and Percent Variation from Nominal, Panel 71-14

while the upper and lower sheet had a combined mean density of 32.2 kg/m^3 (2.01 pcf). The largest variation from the nominal was -22% and was found in Panel 71-14, piece 1-2-2. The lateral density gradients are not as severe with the largest variation from the nominal being +9% and found in Panel 71-14, stack 2-3-X (Table 4-1). Density gradients were checked on eight of twelve panels in the group 71-15 to 71-26. The data indicates that there is a negative density gradient toward the corners of the panels. The middle sheet of the panels also had a lower density. Data from the eight panels is summarized in Table 4-2. The largest single piece variation from the nominal was 31% and occurred in Panel 71-18, piece 2-1-1. Overall, Panel 71-16 had the smallest density gradients while Panel 71-20 ranked second best.

Since the edge pieces from all eight panels had very large density variations, an analysis was made on only the interior pieces. The nominal density is the average density of the interior pieces. The results are given in Table 4-3. In all cases the nominal density of the interior pieces is higher than the nominal density of the full panel.

Six additional 71-panels were cut into small pieces and the density of each calculated to determine the magnitude of longitudinal and lateral density variations. A summary of the density data is presented in Table 4-4. The first four panels listed were all blown with the dichloroethane (DCE) and petroleum ether (SBP) blowing agents and the last two, Panels 71-34 and -36, were blown with only the DCE agent. The 71-36 panel was injection molded while the others were made from rolled millsheet. The nominal densities of the panels ranged from a low of 37.6 kg/m^3 (2.35 pcf) up to a high of 46.1 kg/m^3 (2.88 pcf). The variations in the densities of the three sheets from the nominal panel values ranged from a high of +14 percent on the outer sheets to a low of -15 percent on the inner sheets. In general, the sheets cut from the -34 and -36 panels showed larger variations from the nominal than did the sheets cut from the other four panels. A similar conclusion can be drawn from the data shown in Table 4-4 for the individual pieces. Both the largest positive and negative variations from nominal values occur in pieces cut from Panels 71-34 and -36.

Table 4-1. Maximum Deviations

<u>Panel</u>	<u>Overall Density kg/m³ (pcf)</u>	<u>Max. Longitud. Density Variation From Nominal</u>	<u>Max. Lateral Density Variation From Nominal</u>	<u>Max. Density Variation of any Piece From Nominal</u>
71-12	30.1 (1.88)	12%	+8%	+19%
71-14	28.8 (1.80)	±15%	+9%	-22%

Table 4-2. Density Gradient Summary Sheet

Panel No.	Nominal Density	SHEET						MAXIMUM VARIATION FROM NOMINAL FOR INDIVIDUAL PIECES											
		UPPER		MIDDLE		LOWER		From Upper Sheet			From Middle Sheet			From Lower Sheet					
		ρ	% Var. From Nom.	ρ	% Var. From Nom.	ρ	% Var. From Nom.	Piece	% Var. From Nom.	Piece	% Var. From Nom.	Piece	% Var. From Nom.	Piece	% Var. From Nom.				
71-15	42.29 (2.64)	47.74 (2.98)	13	37.48 (2.34)	-11	46.77 (2.92)	11	431	50.14 (3.13)	19	142	35.56 (2.22)	133	50.14 (3.13)	19				
71-16	38.60 (2.41)	42.13 (2.63)	9	35.24 (2.20)	-9	42.13 (2.63)	9	421	45.33 (2.83)	17	112	31.88 (1.99)	323	45.65 (2.85)	19				
71-17	42.13 (2.63)	47.09 (2.94)	12	36.04 (2.25)	-15	49.18 (3.07)	17	321	49.18 (3.07)	17	442	32.52 (2.03)	423	50.94 (3.18)	21				
71-18	42.77 (2.67)	50.78 (3.17)	19	40.85 (2.55)	-5	51.10 (3.19)	19	211	56.07 (3.50)	31	442	30.76 (1.92)	313	51.74 (3.23)	21				
71-20	49.82 (3.11)	53.02 (3.31)	6	44.69 (2.79)	-10	55.74 (3.48)	12	331	55.10 (3.44)	11	442	40.95 (2.55)	233	58.63 (3.66)	18				
71-22	45.17 (2.82)	49.82 (3.11)	10	39.41 (2.46)	-13	49.66 (3.19)	10	331	51.58 (3.22)	14	412	34.44 (2.15)	343	50.78 (3.17)	13				
71-25	46.93 (2.93)	51.58 (3.22)	10	42.13 (2.63)	-10	55.74 (3.48)	19	331	52.38 (3.27)	12	142	39.41 (2.46)	323	56.23 (3.57)	20				
71-26	47.90 (2.99)	52.86 (3.30)	10	44.05 (2.75)	-8	51.26 (3.20)	7	231	54.62 (3.41)	14	112	40.69 (2.54)	233	53.02 (3.31)	11				

Table 4-3. Density Gradient Summary Sheet on Twelve Interior Pieces Only,
Nominal Density Based on Twelve Pieces

Panel No.	Nominal Density	SHEET												MAXIMUM VARIATION FROM NOMINAL FOR INDIVIDUAL PIECES											
		UPPER				MIDDLE				LOWER				From Upper Sheet			From Middle Sheet			From Lower Sheet					
		ρ	% Var. From Nom.	ρ	% Var. From Nom.	ρ	% Var. From Nom.	ρ	% Var. From Nom.	ρ	% Var. From Nom.	ρ	% Var. From Nom.	Piece	% Var. From Nom.	Piece	% Var. From Nom.	ρ	Piece	% Var. From Nom.	Piece	% Var. From Nom.	ρ	Piece	% Var. From Nom.
71-15	45.01 (2.81)	48.38 (3.02)	+7	39.25 (2.45)	-13	47.42 (2.96)	+5	321	49.02 (2.96)	9	232	38.60 (2.41)	-14	223	47.74 (2.98)	6									
71-16	41.97 (2.62)	43.73 (2.73)	+4	37.32 (2.33)	-11	44.85 (2.80)	+7	321	41.37 (2.77)	6	222	36.34 (2.30)	-12	323	45.63 (2.95)	9									
71-17	45.65 (2.85)	48.38 (3.02)	+6	37.30 (2.36)	-17	50.46 (3.15)	+11	321	49.18 (3.07)	8	332	37.32 (2.33)	-18	233	50.94 (3.18)	12									
71-18	45.97 (2.87)	51.26 (3.20)	+12	38.12 (2.38)	-17	48.38 (3.02)	+5	221	42.22 (3.26)	14	332	37.64 (2.35)	-18	223	40.18 (3.07)	7									
71-20	53.50 (3.34)	54.46 (3.40)	+2	47.42 (2.96)	-11	58.15 (3.63)	+9	331	55.10 (3.44)	3	332	46.93 (2.93)	-12	233	58.63 (3.66)	10									
71-22	47.42 (2.96)	50.78 (3.17)	+7	38.44 (2.40)	-19	49.82 (3.11)	+5	331	51.58 (3.22)	9	332	41.33 (2.58)	-13	223	50.46 (3.15)	6									
71-25	50.14 (3.13)	51.90 (3.24)	+4	42.93 (2.65)	-14	55.74 (3.48)	+11	331	52.38 (3.27)	4	332	42.13 (2.60)	-16	323	56.23 (3.51)	12									
71-26	50.78 (3.17)	54.30 (3.39)	+7	45.33 (2.83)	-11	52.54 (3.28)	+3	231	54.62 (3.41)	8	232	45.01 (2.81)	-11	233	53.02 (3.31)	4									

Table 4-4. Summary of Density Variations Within Panels

Panel No.	Nominal Density	SHEET						MAXIMUM VARIATION FROM NOMINAL FOR INDIVIDUAL PIECES					
		UPPER		MIDDLE		LOWER		From Upper Sheet		From Middle Sheet		From Lower Sheet	
		ρ	% Var. From Nom.	ρ	% Var. From Nom.	ρ	% Var. From Nom.	Piece	% Var. From Nom.	Piece	% Var. From Nom.	Piece	% Var. From Nom.
71-27	40.2 (2.51)	42.8 (2.67)	6	35.5 (2.22)	-12	43.9 (2.74)	9	331 47.5 (2.97)	19	112 34.3 (2.14)	-15	333 47.1 (2.94)	17
71-28	38.9 (2.43)	42.1 (2.63)	8	33.8 (2.11)	-13	41.8 (2.61)	8	121 46.1 (2.88)	19	342 33.1 (2.07)	-15	123 44.5 (2.78)	14
71-29	38.1 (2.38)	42.1 (2.63)	11	34.6 (2.16)	-9	41.6 (2.60)	9	231 44.2 (2.76)	16	312 33.0 (2.06)	-14	143 44.5 (2.78)	17
71-32	45.8 (2.86)	51.8 (3.24)	13	40.7 (2.54)	-11	50.8 (3.17)	11	231 54.8 (3.42)	20	312 38.3 (2.39)	-17	123 52.5 (3.28)	15
71-34	46.1 (2.88)	51.6 (3.22)	12	39.7 (2.48)	-14	51.7 (3.23)	12	321 54.0 (3.37)	17	132 37.0 (2.31)	-20	323 53.3 (3.33)	16
71-36	37.6 (2.35)	42.6 (2.65)	13	32.0 (2.00)	-15	42.9 (2.68)	14	331 44.0 (2.75)	17	412 28.3 (1.77)	-25	233 45.0 (2.81)	20

Also, the two panels blown with DCE ranked sixth and eight best out of the eight panels subjected to thermal performance testing. The panels blown with DCE are clearly inferior to the panels blown with the combination of DCE and SBP both in terms of uniformity of internal structure and overall thermal performance.

A total of seventeen 5 cm (2-inch) thick panels were sliced into sheets and then cut into small pieces for density variation analysis. Of these seventeen, 11 were manufactured with the DCE/CNU combination of blowing agents, four with the DCE/SBP combination, and two with only DCE blowing agent. In general the DCE/CNU panels exhibited the least average density variation from the nominal values, the DCE/SBP panels only slightly higher, and the two DCE panels exhibited considerably higher average density variations than the other panels tested. It should be noted that this same ranking applies to the thermal conductivity results where the DCE/CNU panels were clearly the best. The clear superiority of the DCE/CNU blowing agent combination with regard to uniformity and thermal performance was the primary factor resulting in its selection for use in a follow-on PPO foam order.

Lateral density measurements were made on representative samples of the follow-on PPO foam order. Nine 10.2 x 10.2 cm (4x4 in.) pieces, Figure 4-7, were cut from each of ten panels. Table 4-5 summarizes the results of the density measurements made on each panel. The largest lateral density variation measured was 10%.

Table 4-5. Summary of Lateral Density Variations, 72-Panels

Panel No.	Original Thickness cm (in.)	Test Thickness cm (in.)	Nominal Density kg/m ³ (lb/ft ³)	Maximum Lateral Variation From Nominal %
72-1U	7.62 (3)	2.54 (1.00)	28.49 (1.78)	-7
72-3	7.62 (3)	7.14 (2.81)	25.25 (1.58)	+5
72-18	2.54 (1)	2.54 (1.00)	28.11 (1.75)	+9
72-29	5.08 (2)	4.60 (1.81)	38.65 (2.41)	-6
72-32U	5.08 (2)	1.50 (.59)	44.49 (2.78)	+5
72-34U	5.08 (2)	.76 (.30)	46.23 (2.89)	-5
72-41U	7.62 (3)	2.54 (1.00)	40.92 (2.55)	-4
72-55	2.54 (1)	2.51 (.99)	40.94 (2.56)	-10
72-60	2.54 (1)	2.45 (.98)	50.07 (3.13)	-10
72-74U	7.62 (3)	2.45 (.98)	61.19 (3.82)	-8

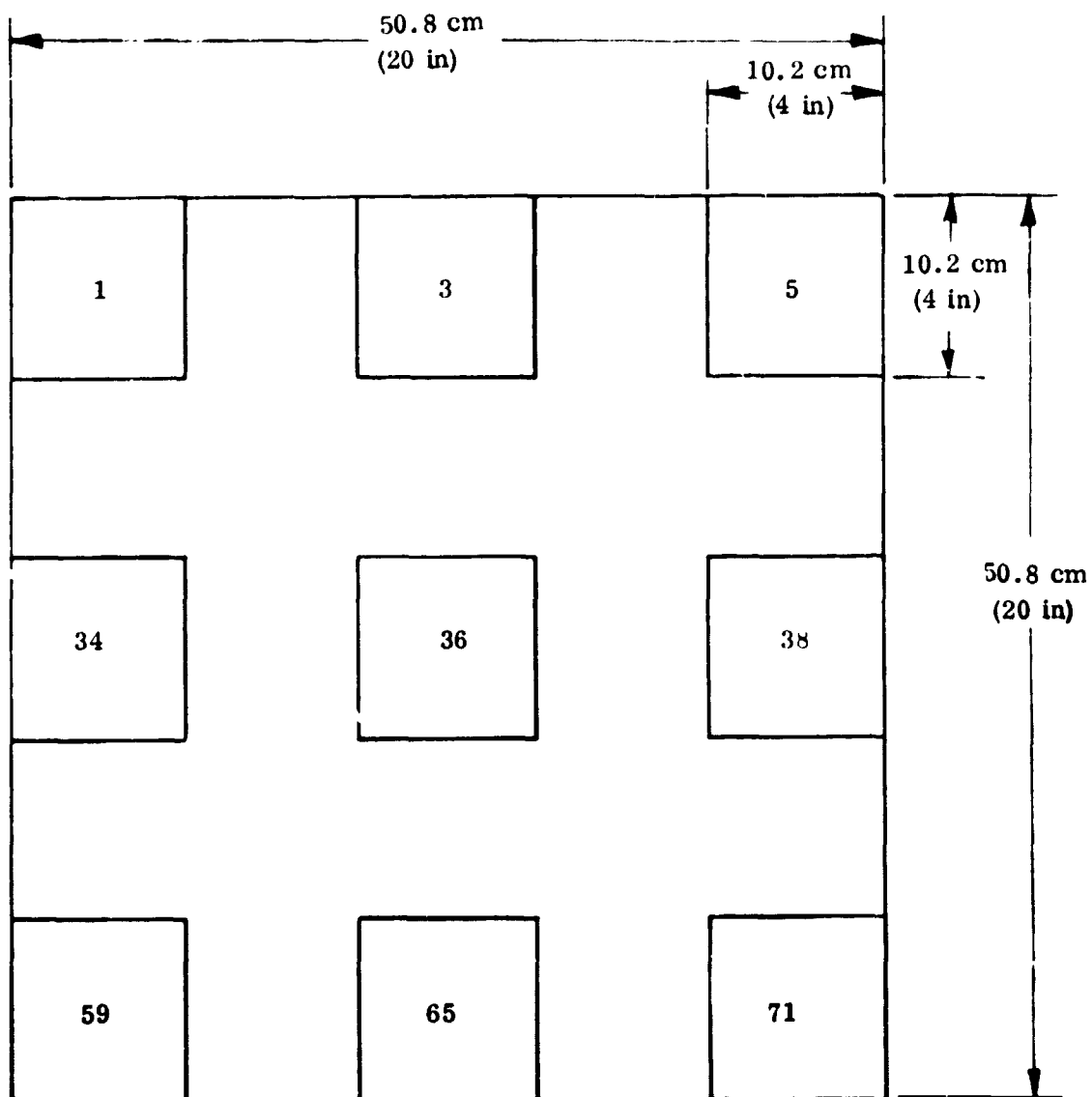


Figure 4-7. Identification of Cut Density Specimens for 72 - Panels

4.1.2 FINAL PRODUCTION PANELS. Ten PPO foam panels were received from the manufacturer, Plastics and Rubber Institute TNO, Delft, Holland, in April 1975. The panels were manufactured to the specification in Section 2.1 of this report. An inventory of these ten panels is listed in Appendix A including ingredients and configuration data. These resin sheets were mixed with DCE/CNU blowing agent, VER nucleating agent and blown from a single rolled mill sheet in a closed press. The panel quality is excellent. Density gradients were measured in two of the panels, 75-7 and 75-9, by Convair and in one of the panels, 75-8 by TNO. Dimensional and density gradient data are listed in Tables 4-6, 4-7, 4-8. Dimensions and longitudinal density gradients are within specification limits. A few pieces appear to be outside the lateral density gradient limit of $\pm 5\%$. Those which do not meet the tolerance limit are always on the low side. The deviation of a subpanel is based on the ρ_k density value of a full size piece before it is cut into ninths (see Figure 4-1). If the allowable $\pm 5\%$ deviation is based on the average density of the nine subpanels after cutting, then the lateral gradients are within the specified limits. An investigation into this phenomena revealed that piece number 3,3,3 from panel number 75-9 weighed 28.1 gm (0.062 lbs) just after cutting and 27.3 gm (0.060 lbs) after 30 days which included some handling. The conclusion is that loose particulate resin dislodged from the subpanel in sufficient quantity to show a measurable weight reduction. Visible quantities can be dislodged by tapping on a table top. This loose material does not however affect the thermal or mechanical quality of the foam material.

4.2 MECHANICAL STRENGTH

The mechanical strength of PPO foam has been evaluated at temperatures of 21K (-422F), 294K (70F) and 422 K (300F) for a wide range of foam densities. Strength tests were performed parallel to the foam cells and perpendicular to the foam cells in both tension and compression. The results of the strength tests have been correlated with foam density.

The strength tests were performed according to ASTM C297. For the parallel tests, 5.08 cm (2 in) square by thick PPO foam blocks were used. The perpendicular specimens were 2.54 cm (1 in) high by 5.08 cm (2 in) long by thick. In the compression tests, the foam blocks were placed between two parallel hardened steel blocks. The tensile specimens were bonded to aluminum blocks, then pinned into fixtures gim-balled in two directions to insure that only axial loading resulted. The specimens tested at 294 K (70F) and 422K (300F) were bonded using Epon 934 epoxy adhesive, specimens tested at 21K (-422F) were bonded using Crest 7343 polyurethane adhesive. All tensile failures occurred in the foam. The test temperatures were controlled by two methods. The specimens tested at 21K (-422F) were submerged in liquid hydrogen for five minutes before starting the test. The tests at 422K (300F) were run in a Missimers chamber. Specimen temperature, was measured with a thermocouple, was held at 422K (300F) for ten minutes before testing.

Table 4-6. PPO Foam Panel 75-7 Density Gradients

Panel No. 75-7

1. Specified Data for Trimmed Panel

$L_s = 70$ cm, $W_s = 60$ cm, $t_s = 47.5$ mm, $\rho_s = 40$ kg/m³

2. Trimmed Panel Measurements

	() _s - 5 mm	() _m	() _s + 5 mm	In Spec	Out of Spec
L	69.5	69.9	70.5	X	
W	59.5	59.7	60.5	X	
	() _s - 0.25 mm	() _m	() _s + 0.25 mm		
t	47.25	47.7	47.75	X	
	() _s × (1-0.05)	() _m	() _s × (1+0.05)		
ρ	38.0	42.0	42.0	X	

3. Sheet Measurements (Longitudinal Variation)

	$\rho_m \times (1-0.1)$	ρ_k	$\rho_m \times (1+0.1)$	In Spec	Out of Spec
ρ_m	37.8		46.2	X	
$\rho_{k=1}$		45.9		X	
$\rho_{k=2}$		39.25		X	
$\rho_{k=3}$		44.5		X	

4. Piece Measurements (Lateral Variation)

	$k = 1$		$k = 2$		$k = 3$	
	$\rho_k \times (1-.05) \times (1+.05)$		$\rho_k \times (1-.05) \times (1+.05)$		$\rho_k \times (1-.05) \times (1+.05)$	
ρ_k	43.6	48.2	37.3	41.2	42.3	46.8
		In Out		In Out		In Out
$\rho_{1,1,k}$	45.2	X	37.1	X	42.4	X
$\rho_{2,1,k}$	46.7	X	39.1	X	44.7	X
$\rho_{3,1,k}$	46.3	X	39.1	X	43.8	X
$\rho_{1,2,k}$	46.6	X	37.9	X	43.9	X
$\rho_{2,2,k}$	47.5	X	39.4	X	45.3	X
$\rho_{3,2,k}$	47.0	X	39.2	X	43.9	X
$\rho_{1,3,k}$	45.0	X	37.3	X	43.1	X
$\rho_{2,3,k}$	46.0	X	39.1	X	44.2	X
$\rho_{3,3,k}$	44.7	X	38.4	X	44.3	X
Average	46.1		38.5		44.0	

Table 4-7. PPO Foam Panel 75-8 Density Gradients

Panel No. 75-8

1. Specified Data for Trimmed Panel

$$L_s = 65 \text{ cm}, \quad W_s = 55 \text{ cm}, \quad t_s = 50 \text{ mm}, \quad \rho_s = 40 \text{ kg/m}^3$$

2. Trimmed Panel Measurements

	() _s - 5 mm	() _m	() _s + 5 mm	In Spec	Out of Spec
L	64.5	65.0	65.5	X	
W	54.5	55.0	55.5	X	
	() _s - 0.25 mm	() _m	() _s + 0.25 mm		
t	49.75	49.8 - 50.2	50.25	X	
	() _s × (1-0.05)	() _m	() _s × (1+0.05)		
ρ	38.6	39.7	42.0	X	

3. Sheet Measurements (Longitudinal Variation)

	ρ _m × (1-0.1)	ρ _k	ρ _m × (1 + 0.1)	In Spec	Out of Spec
ρ _m	35.7		43.7		
ρ _{k=1}		43.3		X	
ρ _{k=2}		36.1		X	
ρ _{k=3}		43.4		X	

4. Piece Measurements (Lateral Variation)

	k = 1		k = 2		k = 3	
	ρ _k × (1-.05) × (1+.05)		ρ _k × (1-.05) × (1+.05)		ρ _k × (1-.05) × (1+.05)	
ρ _k	41.1	45.5	34.3	37.9	41.2	45.6
		In Out		In Out		In Out
ρ _{1,1,k}	44.1	X	37.4	X	44.5	X
ρ _{2,1,k}	44.2	X	37.4	X	45.4	X
ρ _{3,1,k}	42.3	X	36.8	X	43.5	X
ρ _{1,2,k}	43.1	X	37.1	X	43.9	X
ρ _{2,2,k}	44.5	X	37.8	X	44.8	X
ρ _{3,2,k}	42.0	X	36.5	X	43.1	X
ρ _{1,3,k}	41.8	X	35.4	X	42.1	X
ρ _{2,3,k}	42.2	X	35.7	X	43.0	X
ρ _{3,3,k}	40.8	X	35.6	X	41.4	X
Average	42.8		36.6		43.6	

Table 4-8. PPO Foam Panel 75-9 Density Gradients

Panel No. 75-9

1. Specified Data for Trimmed Panel

$L_S = 70 \text{ cm}$, $W_S = 60 \text{ cm}$, $t_S = 47.5 \text{ mm}$, $\rho_S = 40 \text{ kg/m}^3$

2. Trimmed Panel Measurements

	() _S - 5 mm	() _m	() _S + 5 mm	In Spec	Out of Spec
L	69.5	69.8	70.5	X	
W	59.5	59.6	60.5	X	
	() _S - 0.25 mm	() _m	() _S + 0.25 mm		
t	47.25	47.37	47.75	X	
	() _S × (1-0.05)	() _m	() _S × (1+0.05)		
ρ	38.0	40.1	42.0	X	

3. Sheet Measurements (Longitudinal Variation)

	ρ _m × (1-0.1)	ρ _k	ρ _m × (1+0.1)	In Spec	Out of Spec
ρ _m	36.1		44.1		
ρ _{k=1}		43.2		X	
ρ _{k=2}		37.7		X	
ρ _{k=3}		43.0		X	

4. Piece Measurements (Lateral Variation)

	k = 1		k = 2		k = 3	
	ρ _k × (1-.05) × (1+.05)		ρ _k × (1-.05) × (1+.05)		ρ _k × (1-.05) × (1+.05)	
ρ _k	41.0	45.3	35.8	39.6	40.8	45.1
		In Out		In Out		In Out
ρ _{1,1,k}	40.9	X	36.1	X	41.7	X
ρ _{2,1,k}	41.9	X	36.8	X	42.9	X
ρ _{3,1,k}	40.6	X	35.4	X	40.5	X
ρ _{1,2,k}	43.6	X	38.0	X	43.7	X
ρ _{2,2,k}	44.3	X	38.5	X	44.6	X
ρ _{3,2,k}	42.4	X	36.7	X	41.8	X
ρ _{1,3,k}	43.3	X	38.2	X	43.0	X
ρ _{2,3,k}	43.6	X	38.5	X	43.5	X
ρ _{3,3,k}	42.0	X	36.4	X	40.7	X
Average	42.5		37.2		42.5	

Table 4-9. Parallel Tensile Strength of PPO Foam

Panel No. Temp.	Strength, kN/m ² (psi)									
	72-1U		72-3		72-18		72-29		72-32U	
21K (-422F)	1655	(240)	1103	(160)	1772	(257)	1538	(223)	2089	(303)
	1427	(207)	876	(127)	1669	(242)	1710	(248)	1862	(270)
	<u>1482</u>	<u>(215)</u>	<u>938</u>	<u>(136)</u>	<u>1358</u>	<u>(197)</u>	<u>1296</u>	<u>(188)</u>	<u>1696</u>	<u>(246)</u>
	1521	(221)	972	(141)	1600	(232)	1517	(220)	1882	(273)
Ambient	1213	(176)	703	(102)	1827	(265)	1510	(219)	2158	(313)
	1151	(167)	841	(122)	1896	(275)	1524	(221)	2158	(313)
	<u>1248</u>	<u>(181)</u>	<u>662</u>	<u>(96)</u>	<u>1937</u>	<u>(281)</u>	<u>1620</u>	<u>(235)</u>	<u>2151</u>	<u>(312)</u>
	1207	(175)	731	(106)	1889	(274)	1551	(225)	2158	(313)
422K (+300F)	614	(89)	365	(53)	911	(138)	745	(108)	972	(141)
	538	(78)	372	(54)	876	(127)	696	(101)	1048	(152)
	<u>607</u>	<u>(88)</u>	<u>352</u>	<u>(51)</u>	<u>896</u>	<u>(130)</u>	<u>710</u>	<u>(103)</u>	<u>1034</u>	<u>(150)</u>
	586	(85)	359	(52)	903	(131)	717	(104)	1020	(148)

Panel No. Temp.	Strength, kN/m ² (psi)									
	72-34U		72-41U		72-55		72-60		72-74U	
21K (-422F)	2751	(399)	1669	(242)	1772	(257)	1572	(228)	3165	(459)
	2565	(372)	1786	(259)	1669	(242)	1262	(183)	3137	(455)
	<u>2579</u>	<u>(374)</u>	<u>1917</u>	<u>(278)</u>	<u>1358</u>	<u>(197)</u>	<u>1475</u>	<u>(214)</u>	<u>2861</u>	<u>(415)</u>
	2634	(382)	1793	(260)	1600	(232)	1434	(208)	3054	(443)
Ambient	2317	(336)	1620	(235)	1827	(265)	1227	(178)	2613	(379)
	2275	(330)	1731	(251)	1896	(275)	1207	(175)	2579	(374)
	<u>2372</u>	<u>(344)</u>	<u>1572</u>	<u>(228)</u>	<u>1937</u>	<u>(281)</u>	<u>1207</u>	<u>(175)</u>	<u>2710</u>	<u>(393)</u>
	2324	(337)	1641	(238)	1889	(274)	1213	(176)	2634	(382)
422K (+300F)	1117	(162)	979	(142)	911	(138)	552	(80)	359	(52)
	1117	(162)	1062	(154)	876	(127)	531	(77)	1338	(194)
	<u>1069</u>	<u>(155)</u>	<u>876</u>	<u>(127)</u>	<u>896</u>	<u>(130)</u>	<u>579</u>	<u>(84)</u>	<u>862</u>	<u>(125)</u>
	1103	(160)	972	(141)	910	(132)	552	(80)	855	(124)

The results of the mechanical strength tests are summarized in Tables 4-10 through 4-12. Since the foam specimens do not fail in perpendicular compression, the tests were stopped at 20% deformation and the yield strength taken at a 2% offset. Typical stress failures are shown in Figures 4-8 through 4-10. Figure 4-8 shows the two locations where parallel compressive failures occurred. The compressive failures occur in the low density middle section of the foam; which is the center of a normal panel on the right side of Figure 4-8 and the lower surface of the piece cut from the outer third of a large panel (specimen on the left side of Figure 4-8). Figure 4-9 shows perpendicular (specimen on the left) and parallel (specimen on the right) tensile failures. Figure 4-10 compares a perpendicular tension specimen tested at 294K (70F) with a similar specimen from the same foam panel tested at 422K (300F). Note that the specimen run at 422K (300F) elongated with a reduced cross-section while specimens run at the lower temperatures has a constant cross-section.

Figure 4-11 shows the strength of PPO foam as a function of density. The two low parallel tension data points at a density of 50.0 kg/m^3 (3.13 lb/ft^3) are from panel 72-60 which had a severely curved cell structure resulting in reduced parallel tensile strength. Density gradients through the foam test specimens results in a scatter of the strength data. The strength of PPO foam increases with increasing density. Figure 4-12 shows the strength of PPO foam as a function of temperature. Parallel tension and compression and perpendicular tension and compression are shown. The strength decreases with increasing temperature with an upper usable limit near 422K (300F).

Table 4-10. Parallel Compressive Strength of PPO Foam

Panel No. Temp.	Strength, kN/m ² (psi)									
	72-1U		72-3		72-18		72-29		72-32U	
21K (-422F)	296	(43)	159	(23)	248	(36)	986	(143)	1027	(149)
	310	(45)	159	(23)	407	(59)	917	(133)	1110	(161)
	<u>269</u>	<u>(39)</u>	<u>234</u>	<u>(34)</u>	<u>283</u>	<u>(41)</u>	<u>1000</u>	<u>(145)</u>	<u>1145</u>	<u>(166)</u>
	290	(42)	186	(27)	310	(45)	965	(140)	1096	(159)
Ambient	207	(30)	145	(21)	179	(26)	621	(90)	800	(116)
	207	(30)	152	(22)	200	(29)	641	(93)	855	(124)
	<u>207</u>	<u>(30)</u>	<u>145</u>	<u>(21)</u>	<u>255</u>	<u>(37)</u>	<u>641</u>	<u>(93)</u>	<u>855</u>	<u>(124)</u>
	207	(30)	145	(21)	214	(31)	634	(92)	834	(121)
422K (+300F)	159	(23)	110	(16)	103	(15)	421	(61)	490	(71)
	145	(21)	110	(16)	117	(17)	441	(64)	517	(75)
	<u>159</u>	<u>(23)</u>	<u>103</u>	<u>(15)</u>	<u>110</u>	<u>(16)</u>	<u>421</u>	<u>(61)</u>	<u>524</u>	<u>(76)</u>
	152	(22)	110	(16)	110	(16)	427	(62)	510	(74)

Panel No. Temp.	Strength, kN/m ² (psi)									
	72-34U		72-41U		72-55		72-60		72-74U	
21K (-422F)	1365	(198)	765	(111)	1255	(182)	414	(60)	2000	(290)
	1269	(184)	883	(128)	1131	(164)	434	(63)	1613	(234)
	<u>924</u>	<u>(134)</u>	<u>807</u>	<u>(117)</u>	<u>1379</u>	<u>(200)</u>	<u>586</u>	<u>(85)</u>	<u>1496</u>	<u>(217)</u>
	1186	(172)	820	(119)	1255	(182)	476	(69)	1703	(247)
Ambient	855	(124)	538	(78)	683	(99)	234	(34)	1041	(151)
	855	(124)	538	(78)	827	(120)	310	(45)	1055	(153)
	<u>869</u>	<u>(126)</u>	<u>538</u>	<u>(78)</u>	<u>876</u>	<u>(127)</u>	<u>669</u>	<u>(97)</u>	<u>986</u>	<u>(143)</u>
	862	(125)	538	(78)	793	(115)	407	(59)	1027	(149)
422K (+300F)	524	(76)	386	(56)	552	(80)	159	(23)	662	(96)
	510	(74)	359	(52)	545	(79)	207	(30)	648	(94)
	<u>545</u>	<u>(79)</u>	<u>359</u>	<u>(52)</u>	<u>476</u>	<u>(69)</u>	<u>172</u>	<u>(25)</u>	<u>648</u>	<u>(94)</u>
	524	(76)	365	(53)	524	(76)	179	(26)	655	(95)

Table 4-11. Perpendicular Tensile Strength of PPO Foam

Panel No. Temp.	Strength kN/m ² (psi)									
	72-1U		72-3		72-18		72-29		72-32U	
21K (-422F)	138	(20)	97	(14)	186	(27)	365	(53)	427	(62)
	172	(25)	69	(10)	179	(26)	207	(30)	359	(52)
	<u>172</u>	<u>(25)</u>	<u>97</u>	<u>(14)</u>	<u>152</u>	<u>(22)</u>	<u>255</u>	<u>(37)</u>	<u>269</u>	<u>(39)</u>
	159	(23)	90	(13)	172	(25)	276	(40)	352	(51)
Ambient	193	(28)	138	(20)	179	(26)	310	(45)	421	(61)
	193	(28)	138	(20)	186	(27)	310	(45)	310	(45)
	<u>193</u>	<u>(28)</u>	<u>145</u>	<u>(21)</u>	<u>172</u>	<u>(25)</u>	<u>290</u>	<u>(42)</u>	<u>448</u>	<u>(65)</u>
	193	(28)	138	(20)	179	(26)	303	(44)	393	(57)
422K (+300F)	117	(17)	76	(11)	103	(15)	338	(49)	207	(30)
	103	(15)	69	(10)	90	(13)	352	(51)	186	(27)
	<u>103</u>	<u>(15)</u>	<u>69</u>	<u>(10)</u>	<u>83</u>	<u>(12)</u>	<u>386</u>	<u>(56)</u>	<u>228</u>	<u>(33)</u>
	110	(16)	69	(10)	90	(13)	359	(52)	207	(30)

Panel No. Temp.	Strength, kN/m ² (psi)									
	72-34U		72-41U		72-55		72-60		72-74U	
21K (-422F)	303	(44)	359	(52)	331	(48)	455	(66)	841	(122)
	421	(61)	310	(45)	303	(44)	552	(80)	552	(80)
	-	-	<u>317</u>	<u>(46)</u>	<u>379</u>	<u>(55)</u>	<u>372</u>	<u>(54)</u>	<u>524</u>	<u>(76)</u>
	365	(53)	331	(48)	338	(49)	462	(67)	641	(93)
Ambient	414	(60)	338	(49)	359	(52)	517	(75)	600	(87)
	393	(57)	317	(46)	400	(58)	503	(73)	510	(74)
	<u>386</u>	<u>(56)</u>	<u>317</u>	<u>(46)</u>	<u>345</u>	<u>(50)</u>	<u>490</u>	<u>(71)</u>	<u>572</u>	<u>(83)</u>
	400	(58)	324	(47)	365	(53)	503	(73)	558	(81)
422K (+300F)	117	(17)	179	(26)	186	(27)	234	(34)	221	(32)
	207	(30)	172	(25)	214	(31)	228	(33)	276	(40)
	<u>179</u>	<u>(26)</u>	<u>165</u>	<u>(24)</u>	<u>186</u>	<u>(27)</u>	<u>221</u>	<u>(32)</u>	<u>269</u>	<u>(39)</u>
	165	(24)	172	(25)	193	(28)	228	(33)	255	(37)

Table 4-12. Perpendicular Compressive Strength of PPO Foam

Panel No. Temp.	Strength kN/m^2 (psi)			
	72-1U	72-3	72-18	72-32U
21K (-422F)	41.4 (6.0)	13.8 (2.0)	151.7 (22.0)	133.1 (19.3)
	27.6 (4.0)	10.3 (1.5)	81.4 (11.8)	39.3 (5.7)
	17.9 (2.6)	11.0 (1.6)	-	77.2 (11.2)
	29.0 (4.2)	11.7 (1.7)	116.5 (16.9)	82.7 (12.0)
Ambient	17.9 (2.6)	13.1 (1.9)	15.9 (2.3)	60.7 (8.8)
	13.8 (2.0)	9.0 (1.3)	17.2 (2.5)	46.2 (6.7)
	12.4 (1.8)	11.7 (1.7)	17.2 (2.5)	46.2 (6.7)
	14.5 (2.1)	11.0 (1.6)	16.5 (2.4)	51.0 (7.4)
422K (+300F)	14.5 (2.1)	6.2 (0.9)	7.6 (1.1)	31.7 (4.6)
	6.9 (1.0)	7.6 (1.1)	6.2 (0.9)	51.0 (7.4)
	9.0 (1.3)	7.6 (1.1)	6.9 (1.0)	27.6 (4.0)
	8.3 (1.2)	6.9 (1.1)	6.9 (1.0)	29.6 (4.3)

Panel No. Temp.	Strength kN/m^2 (psi)			
	72-41U	72-55	72-60	72-74U
21K (-422F)	62.7 (9.1)	33.1 (4.8)	139.3 (20.2)	138.6 (20.1)
	85.5 (12.4)	31.0 (4.5)	80.7 (11.7)	151.0 (21.9)
	130.3 (18.9)	57.9 (8.4)	133.8 (19.4)	160.0 (23.2)
	93.1 (13.5)	40.7 (5.9)	117.9 (17.1)	149.6 (21.7)
Ambient	53.8 (7.8)	44.8 (6.5)	106.9 (15.5)	116.5 (16.9)
	47.6 (6.9)	43.4 (6.3)	49.6 (7.2)	111.7 (16.2)
	49.0 (7.1)	55.2 (8.0)	105.5 (15.3)	100.7 (14.6)
	50.3 (7.3)	47.6 (6.9)	86.9 (12.6)	109.6 (15.9)
422K (+300F)	30.3 (4.4)	20.7 (3.0)	36.5 (5.3)	68.9 (10.0)
	29.0 (4.2)	25.5 (3.7)	38.6 (5.6)	71.7 (10.4)
	29.6 (4.3)	31.0 (4.5)	-	73.1 (10.6)
	29.6 (4.3)	25.5 (3.7)	37.2 (5.4)	71.0 (10.3)

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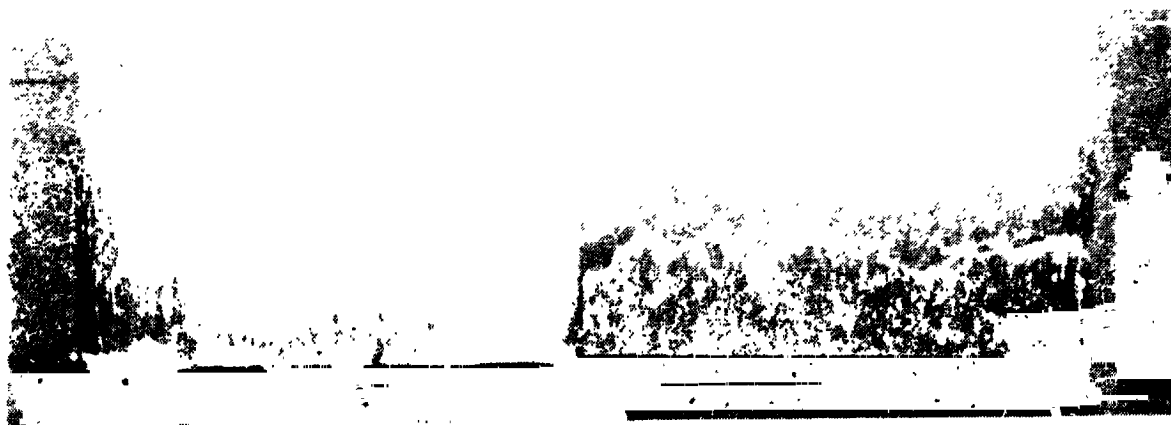


Figure 4-8 . Typical Parallel Compressive Failures in PPO Foam

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PERPENDICULAR TENSION

PARALLEL TENSION

Figure 4-9. Typical Tensile Failures in PPO Foam

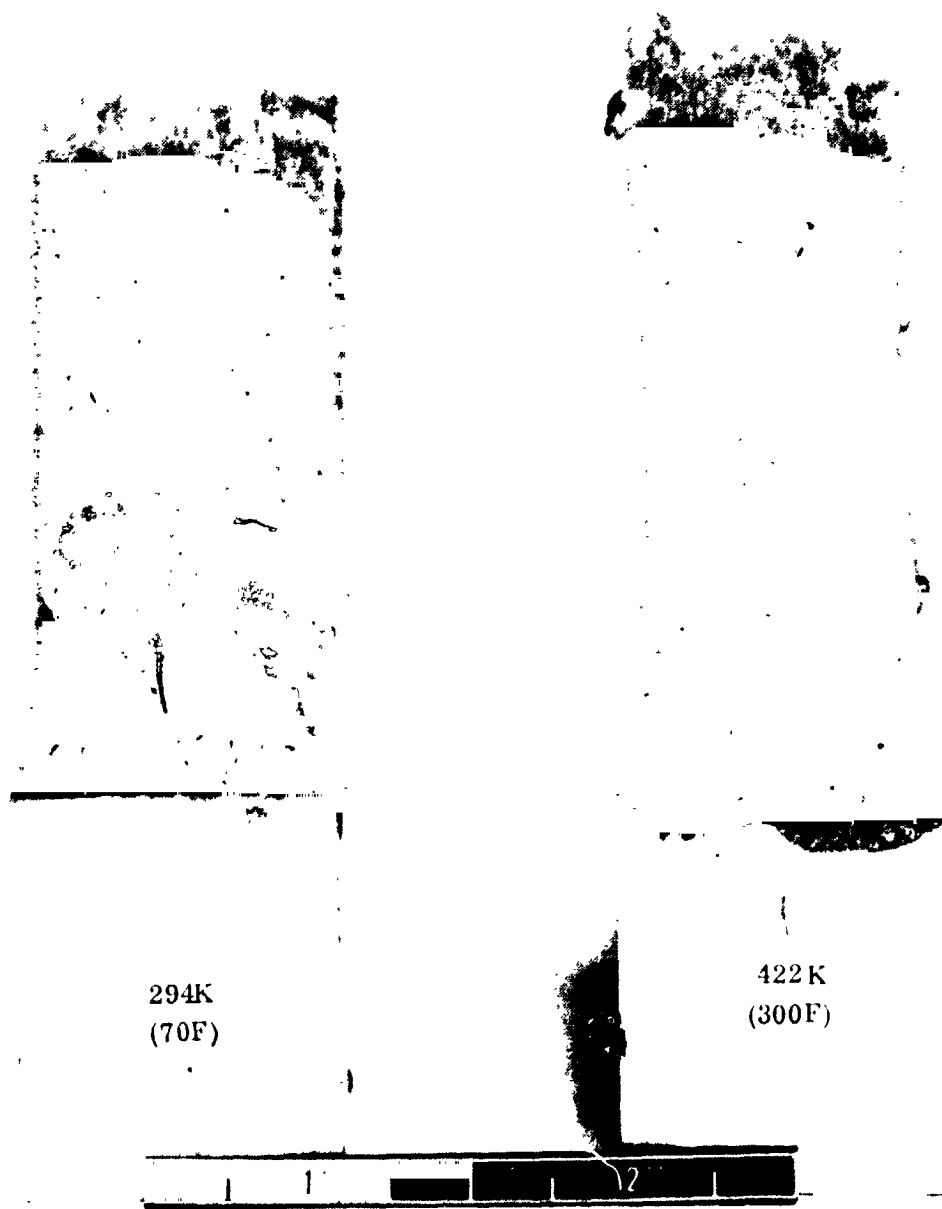


Figure 4-10 Comparison of Perpendicular Tensile Failures in PPO Foam

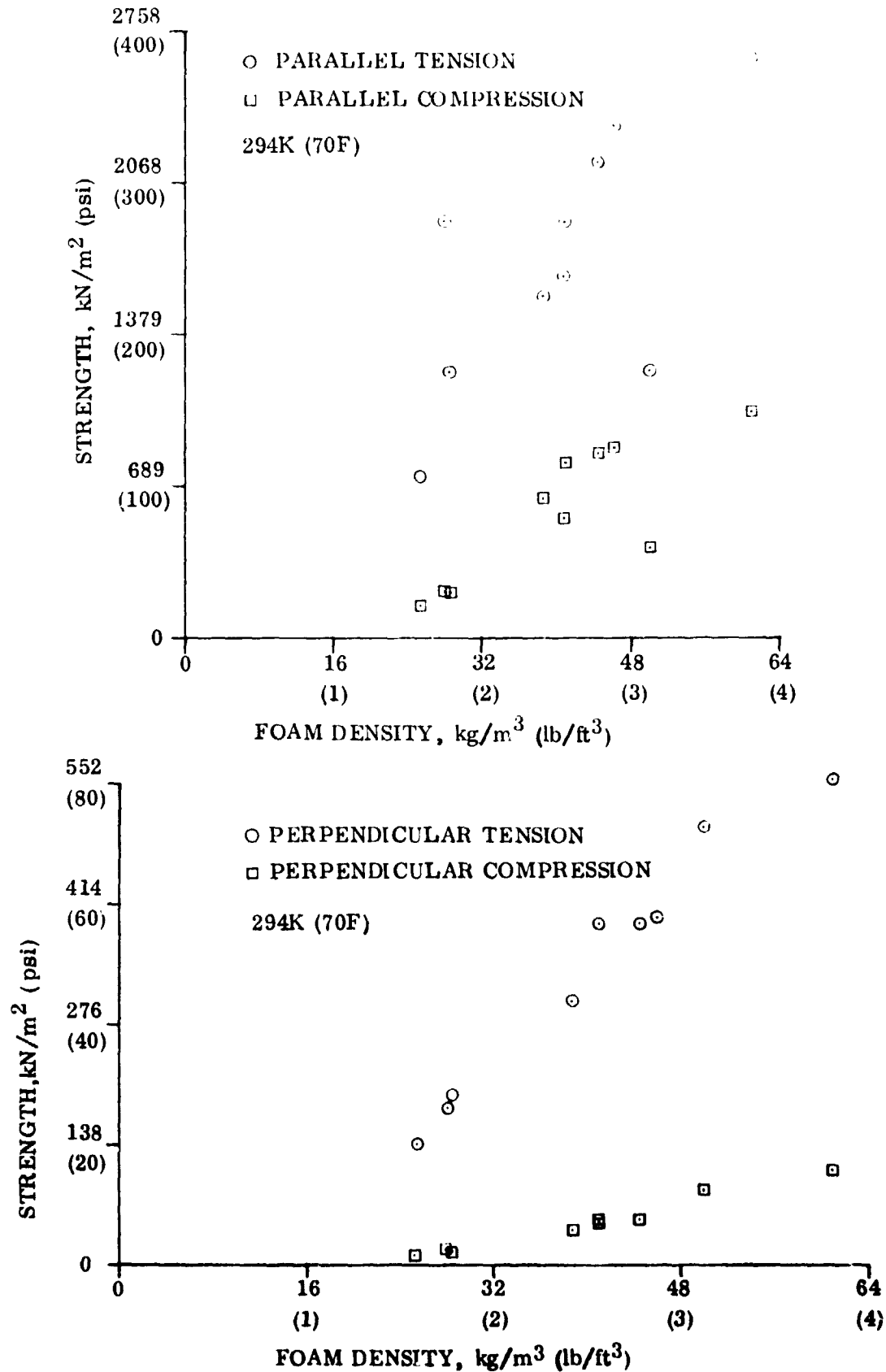


Figure 4-11. Variation of PPO Foam Strength With Foam Density

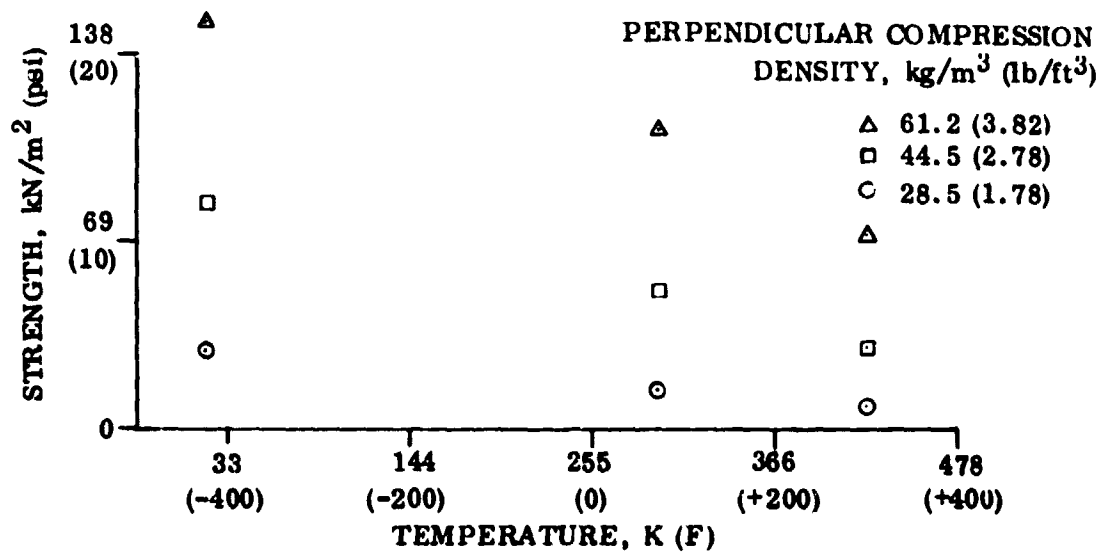
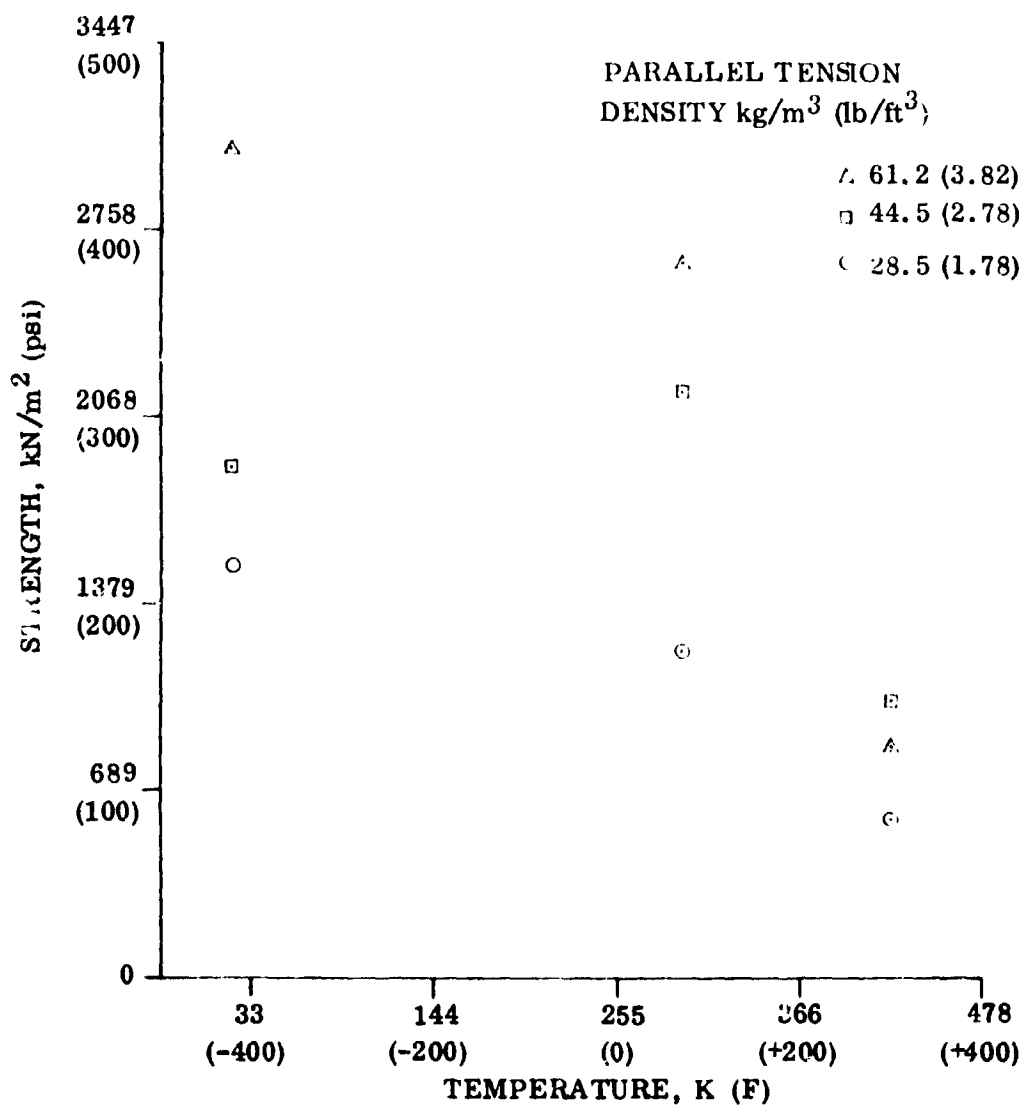


Figure 4-12. Strength of PPO Foam as a Function of Temperature

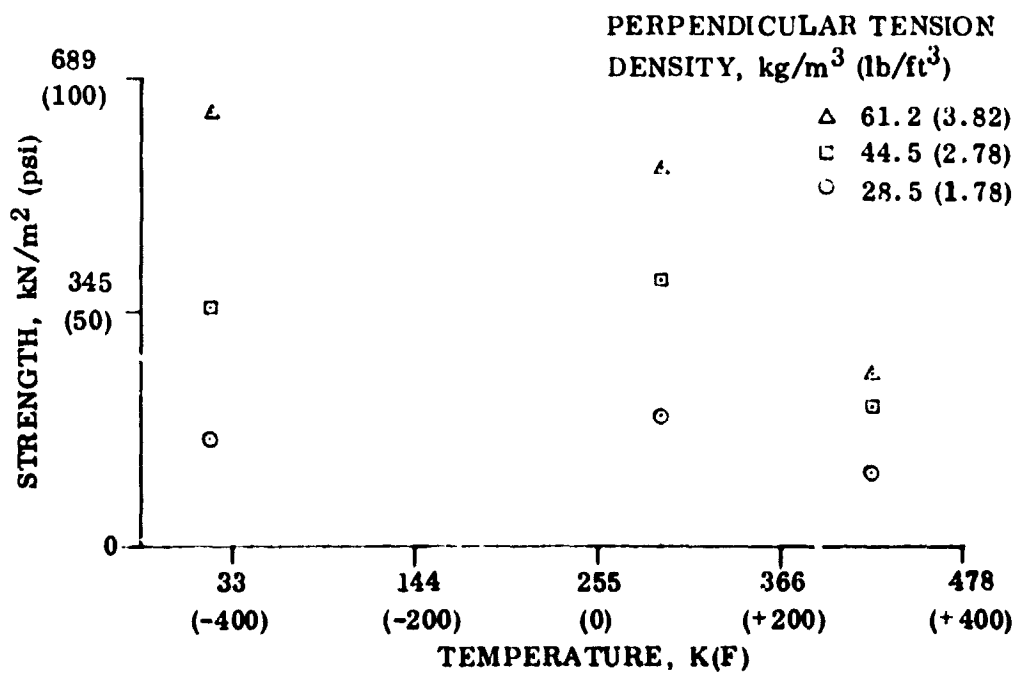
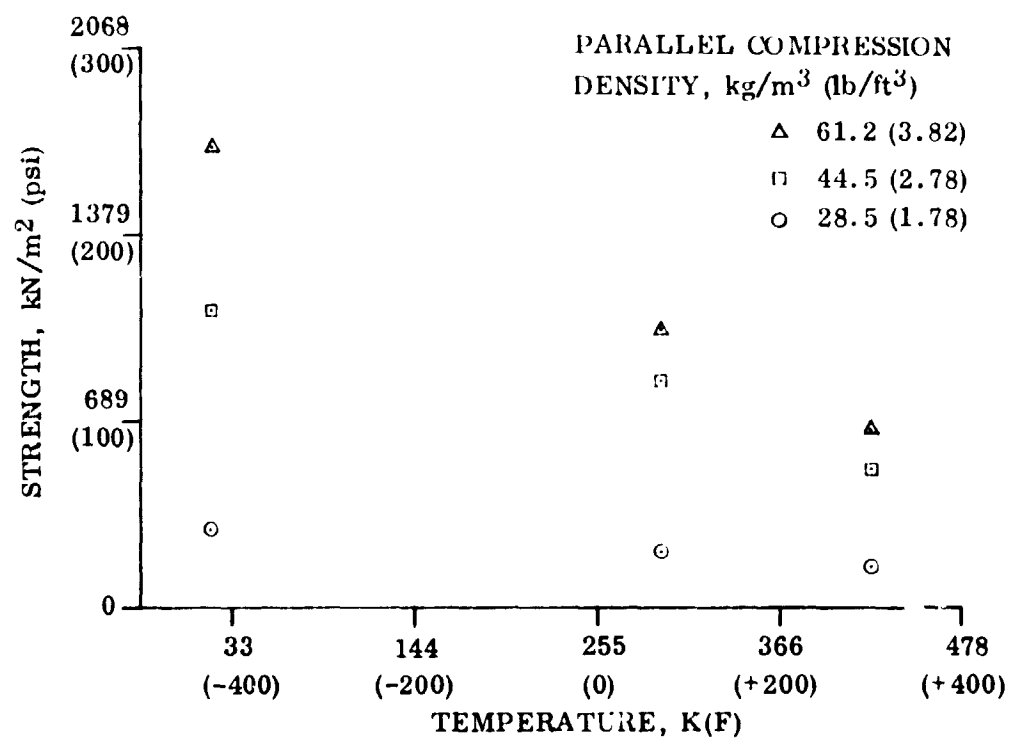


Figure 4-12. Concluded

SECTION 5

THERMAL ENVIRONMENT TESTS

5.1 THERMAL SOAK

During the course of a Space Shuttle mission cycle, the internal insulation system of the liquid hydrogen propellant tanks will be subjected to severe extremes of thermal environments. The most critical phase of the mission, from the standpoint of the insulation system, will most likely be atmospheric entry where the empty propellant tanks will be heated to a temperature as high as 450K (350F). The insulation material must be capable of withstanding repeated exposures to this environment with a minimum of permanent dimensional and weight change and with a minimal reduction in mechanical strength.

PPO foam specimens, taken from panels blown with dichloroethane, have been subjected to long term exposure at elevated temperatures in various gas environments to evaluate the effect on material physical characteristics. Eighteen foam specimens, $7.6 \times 12.7 \times 4.6$ cm ($3 \times 5 \times 1.8$ in.), were weighed and measured, installed in three purge bags (6 in each bag) and placed in a circulating air oven. The three bags were purged with gaseous helium, nitrogen, and air, respectively. The oven was heated to 450K (350F) and the samples were withdrawn from the bags according to a predetermined schedule. The time at 450K (350F) and the resulting weight and volume changes for each specimen are shown in Table 5-1. Weight changes were typically less than one percent and were generally negative for the samples purged with GHe and GN₂ and positive for the samples purged with air. All of the specimens shrunk during the exposure with volume changes ranging from -1 to -4.4 percent. The resulting density increases for the 18 specimens ranged from 0.4 to 5.6 percent.

Table 5-1. PPO Foam Thermal Soak at 450K (350F)

Spec. No.	Time at 450K (350F) (hrs)	Weight Change			Volume Change		
		GHe (%)	GN ₂ (%)	Air (%)	GHe (%)	GN ₂ (%)	Air (%)
1	2.7	+0.3	-0.3	0	-1.5	-2.3	-2.4
2	6.0	-0.6	0	-0.3	-1.0	-2.6	-0.6
3	9.5	-0.6	-0.6	0	-1.0	-1.4	-2.4
4	24.25	+2.5	-0.3	+0.3	-2.7	-2.7	-4.0
5	28.25	-0.3	-0.7	+0.6	-3.1	-2.4	-4.4
6	28.25	-0.6	-0.3	+0.6	-4.1	-2.2	-3.1

As shown in Figure 5-1, the samples soaked in air became progressively darker with exposure to the 450K (350F) environment indicating that an oxidation process was occurring. The other 12 samples soaked in helium and nitrogen showed no discoloration after exposure at the same temperature and times.

A total of five separate thermal aging test runs were made in the laboratory series of tests. In the first three, specimens of PPO foam were sealed in glass tubes in specified gas atmospheres and aged at 450K (350F) for various periods of time. Both the foam and the gas atmospheres were analyzed for evidence of deterioration of the foam. The test conditions and procedures for these three runs are shown in Tables 5-2, -3, and -4. In the fourth test run, a weighed and measured block of foam was aged in a hydrogen atmosphere in a steel bomb at elevated temperature. At specified times the hydrogen atmosphere was analyzed for evidence of foam deterioration, and the foam block was reweighed and remeasured. At the end of the test the foam block was exposed to the ambient air for 48 hours and again measured and weighed. The procedure and conditions for this test run are given in Table 5-5.

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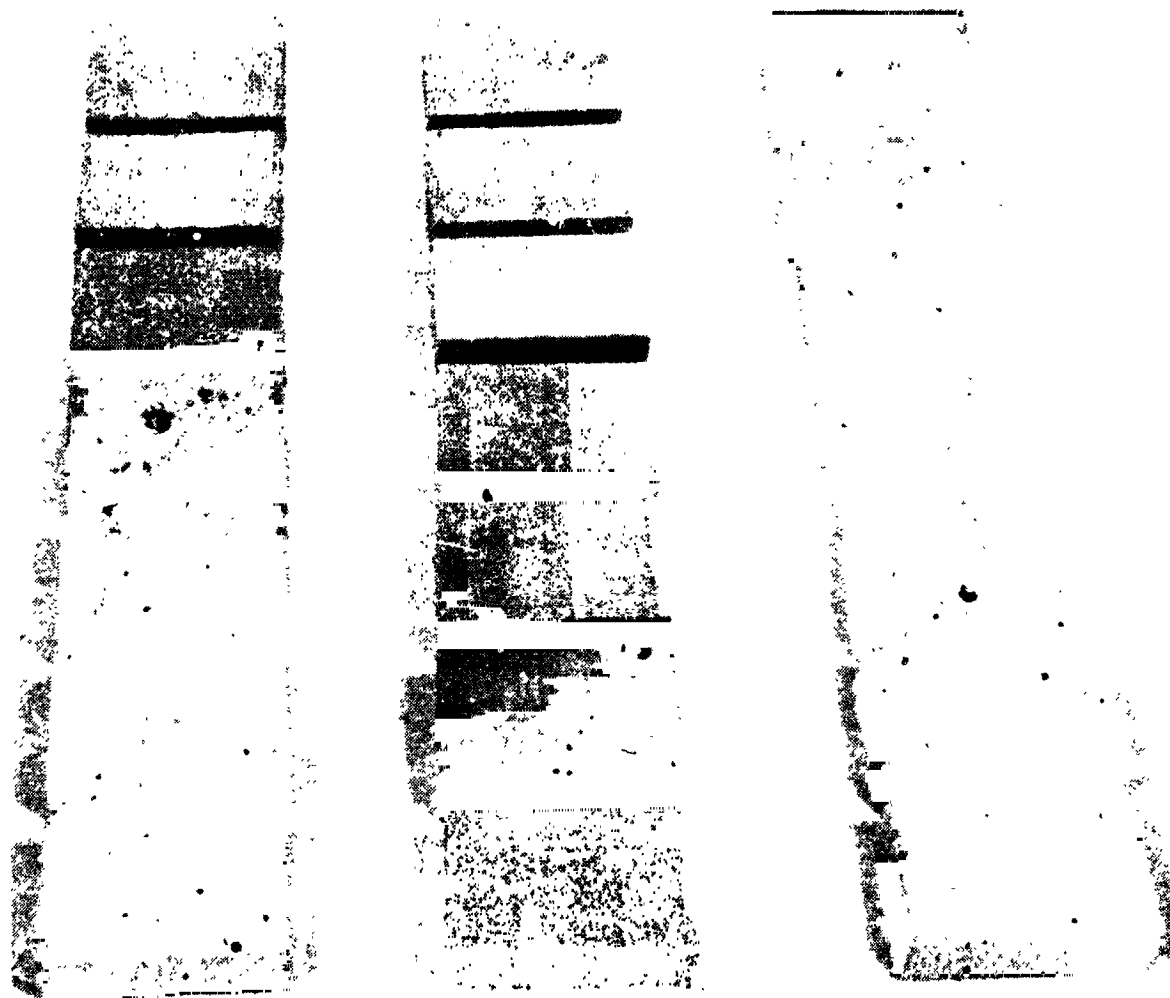


Figure 5-1. Thermal Soak Specimens

Table 5-2. Conditions for Thermal Soak Test Run No. 1

Test Duration	Temperature	Sample No.	Preparation	
			Evacuate to 1 mm Pressure	Backfill With
48 hrs	450K (350F)	A	+	H ₂
		B	+	He
		C	+	N ₂
		D	+	Air
		E	-	Air

Procedure: Obtain five glass cylinders approximately 1 cm (.4 in) diameter by 15 cm (5.9 in) long. Seal one end of the cylinder and allow to cool. Cut PPO foam specimens approximately 0.7 cm (0.3 in) diameter by 4.5 cm (1.8 in) long and place in the tubes. Evacuate tubes A through D to 1 mm pressure and backfill with the test gas. Seal the open ends of the tubes. Place tubes in oven at 450K (350F) for 49 hours. Remove and cool. Run infrared and mass spectrometer analyses on the gas in the tubes. Run infrared analyses on the PPO foam specimens.

Table 5-3. Conditions for Thermal Soak Test Run No. 2

Test Duration	Temp.	Sample No.	Preparation			
			Evacuate to 1 mm Pressure	Backfill With	Evacuate to 1 mm Pressure	Backfill With
24 hrs	450K (350F)	A	+	H ₂	+	H ₂
		B	+	He	+	He
		C	+	N ₂	+	N ₂
		D	+	Dry Air	-	-
		E	-	Amb. Air	-	-
		F	+	-	-	-

Procedure: Obtain six glass cylinders approximately 2 cm (0.8 in) dia by 15 cm (5.9 in) long, with vacuum stopcocks on one end. Cut PPO foam specimens approximately 1.5 cm (0.6 in) dia by 4.5 cm (1.8 in) long and place in the glass tubes. Evacuate tubes (except tubes D and E) to 1 mm (0.04 in) pressure and backfill (except tube F) with the test gas. Repeat the evacuation and backfill. Seal the open ends of the tubes. Place the tubes in the oven at 450K (350F) for 24 hours. Remove and cool. Obtain color photograph of the six tubes. Run infrared and mass spectrometer analyses of the gas in the tubes. Run infrared analyses of the PPO foam specimens.

Table 5-4. Conditions for Thermal Soak Test Run No. 3

Test Duration	Temp.	Sample No.	Preparation			
			Evacuate to 1 mm Pressure	Backfill With	Evacuate to 1 mm Pressure	Backfill With
104 hrs	450K (350F)	A	+	H ₂	+	H ₂
		B	+	N ₂	+	N ₂
		C	+	He	+	He
		D	+	Air	+	Air
		E	Purge tube with 15 volumes of GN ₂			
		F	+	-	-	-

Procedure: Obtain six glass cylinders approximately 2 cm (0.8 in) dia by 15 cm (5.9 in) long, and seal one end. Cut PPO foam specimens approximately 1.5 cm (0.6 in) dia by 4.5 cm (1.8 in) long, and place in the tubes. Evacuate tubes (except E) to 1 mm (0.04 in) pressure and backfill. Seal the open ends of the tubes. Obtain color photograph of the six tubes. Place tubes in an oven at 450K (350F) for 104 hours. Obtain a color photograph of the tubes during the test at 24 hours, 48 hours, 72 hours, 96 hours, and 104 hours. Remove the tubes from the oven and cool. Run infrared and mass spectrometer analyses on the gas in the tubes. Run infrared analyses on the PPO foam specimens.

Table 5-5. Conditions for Thermal Soak Test Run No. 4

Test Duration	Temp.	Sample No.	Preparation			
			Evacuate to 1 mm Pressure	Backfill With	Evacuate to 1 mm Pressure	Backfill With
24 hrs	450K (350F)	A	+	H ₂	+	H ₂
48 hrs		B	+	H ₂	+	H ₂
72 hrs		C	+	H ₂	+	H ₂
140 hrs		D	+	H ₂	+	H ₂

Procedure: Obtain accurate dimensions and weight of a block of PPO foam approx. 4.5 × 4.5 × 12.5 cm (1.8 × 1.8 × 4.9 in). Place in steel bomb, evacuate to 1 mm pressure and backfill with hydrogen. Repeat evacuation and backfill. Place in oven at 450K (350F) for 140 hrs. At 24 hrs, 48 hrs and 72 hrs run infrared and mass spectrometer analyses on the gas atmosphere and re-weight the PPO foam specimen. After replacing the specimen in the bomb, evacuate to 1 mm pressure and backfill with hydrogen, twice. At 140 hrs, run infrared and mass spectrometer analyses on the gas in the bomb, and reweigh and remeasure the PPO foam block. Allow the PPO foam block to come to equilibrium in air for 48 hrs, and re-weigh and re-measure the specimen.

Analysis methods consisted of mass spectrometer and infrared absorption analyses of the gas atmospheres, and infrared absorption analysis of the PPO foam samples. Mass spectrometer analysis provides information on the relative amounts of molecular species present in the sample in the gas phase. These analyses were made with a CEC Model 21-130 Mass Spectrometer. Infrared absorption analysis provides information on the chemical bonds present in the sample, which may be solid, liquid, or gas. These analyses were made with a Perkin-Elmer Model 21 Infrared Spectrophotometer.

Table 5-6 gives the significant results of the mass spectrometer analyses for the four test runs. The actual analyses obtained scan over the entire mass range from m/e 2 to m/e 50, but no molecular species were detected other than oxygen, nitrogen, argon, carbon dioxide, and water, and only the oxygen, carbon dioxide, and water were considered significant. The results of Table 5-6 are in terms of relative concentration for the three species water (m/e 18), oxygen (m/e 32), and carbon dioxide (m/e 44). The last entry in the table gives the typical output for a sample of laboratory air as a reference.

Examination of the data shows a proportional relationship in the amounts of carbon dioxide and oxygen. Compare, for example, test runs 2A, B, and C with test runs 2D, E, and F. The data also shows an increased amount of water present in the samples with hydrogen atmospheres. Examples are test runs 1A versus 1B and D, and test runs 2A versus 2B, C and D.

Table 5-7 gives the weight and dimension data from test run 4. The data shows that although no measurable change occurred in the dimensions of the block, a small weight loss, of the order of 0.5%, was detected for the 140 hour run. The foam block did not gain back any appreciable amount of the weight loss when equilibrated with ambient air which indicates that the weight loss was an actual change in the weight of the PPO foam, and not just a loss of absorbed water vapor.

Infrared spectra of the gas atmospheres in the tubes were essentially flat traces indicating that no detectable quantities of IR absorbing species were present in the gases. Specifically, detectable amounts of carbon dioxide and water vapor which would produce absorption bands at 4.5 and 6.0 microns wavelength, respectively, were absent. Infrared analyses were also made of the foam, PPO rod stock, and sample of PPO resin containing the blowing agent, dichloroethane. The latter sample contained an absorption band at 14.1 microns wavelength indicating the presence of the chlorinated hydrocarbon. This band is not present in the spectrum of the foam indicating that the blowing agent is essentially removed from the foam during the blowing process.

Table 5-6. Mass Spectrometer Analyses

	Sample No.	Atmosphere	Mass Number		
			18 H ₂ O	32 O ₂	44 CO ₂
Test 1.	A	H ₂	1.02	2.14	.09
	B	He	.32	7.59	.26
	C	N ₂		Sample Lost	
	D	Air	.21	8.91	.15
	E	Air		Sample Lost	
Test 2.	A	H ₂	.75	.54	.06
	B	He	.34	3.08	.17
	C	N ₂	.13	2.03	.06
	D	Air	.09	8.71	.73
	E	Air	.13	8.45	.78
	F	Vacuum	.33	8.16	.47
Test 3.	A	H ₂	1.09	2.93	.15
	B	N ₂	.14	1.41	.04
	C	He		Sample Lost	
	D	Air		Sample Lost	
	E	N ₂	.45	3.91	.08
	F	Vacuum	.47	8.80	.56
Test 4.	A	H ₂	1.02	.04	.10
	B	H ₂	.55	.02	.06
	C	H ₂	.71	.05	.05
Air Standard			.46	9.53	.09

Units for the data are chart divisions recorder output per micron sample pressure.

Table 5-7. Test Run 4 Results

	<u>Weight</u>	<u>Percent Change</u>	<u>Dimensions</u> ± 0.5 mm (0.02 in)
Initial	9.7715 gm (.02154 lb)	-	46 x 46 x 128 mm. (1.81×1.81×5.04 in)
24 hrs. @ 450K	9.7465 (.02149)	-0.26	46 x 46 x 128 "
48 hrs. @ 450K	9.7368 (.02147)	-0.36	46 x 46 x 128 "
72 hrs. @ 450K	9.7328 (.02146)	-0.40	46 x 46 x 128 "
140 hrs. @ 450K	9.7215 (.02143)	-0.51	46 x 46 x 128 "
48 hrs. ambient	9.7255 (.02143)	-0.50	46 x 46 x 128 "

Those samples aged in an oxidizing atmosphere and one sample aged for one-half hour in air at 505K (450F) showed the presence of a small absorption band at 5.8 to 5.9 microns wavelength. This band represents the presence of a carbonyl group, such as an ester, aldehyde, or ketone functional group, formed by a chemical reaction. The carbonyl band was not present in the spectra of the other samples.

A fifth thermal soak test was performed on PPO foam samples from the master 72 panel order to obtain data relative to changes of the foam under conditions of heat treatment. The heat treatment at 450K (350F) was performed under various gas atmospheres. Instrumental chemical techniques of infrared spectroscopy and mass spectrometry were utilized to obtain test data.

The PPO foam specimens were tested in glass tubes made with a pressure plate. The pressure plate permitted easy introduction of the sample into the analytical instruments for testing. Two weighed cylindrical PPO specimens, approximately 2.5 centimeters long, 1.5 cm in diameter (1 in. \times 0.6 in.) were placed in each of the seven tubes. Twice the tubes were evacuated to 1 mm Hg pressure and backfilled with the respective test gas except in the case of the vacuum specimens E₁ and E₂. Specimens E₁ and E₂ were sealed under vacuum without backfilling. The samples were prepared and tested according to the following designations:

<u>Sample</u>	<u>PPO Foam</u>	<u>Gas</u>
A	Low Density 30 kg /m ³ (1.87 pcf)	Hydrogen gas
B	Low Density 30 kg /m ³ (1.87 pcf)	Helium gas
C	Low Density 30 kg /m ³ (1.87 pcf)	Nitrogen gas
D	High Density 50 kg /m ³ (3.12 pcf)	Nitrogen gas
E ₁	Low Density 30 kg /m ³ (1.87 pcf)	Vacuum
E ₂	Low Density 30 kg /m ³ (1.87 pcf)	Vacuum
F	Low Density 30 kg /m ³ (1.87 pcf)	Ambient Air

The samples were photographed (in their glass tubes) prior to the thermal soak procedure. The samples were placed in an oven at 450°K (350°F). They were removed and photographed after 52 hours. Forty-eight additional hours of thermal soak concluded the test. Additional photos were made of the specimens.

A stopcock tube with a fitting for the mass spectrometer was attached to the glass specimen tube by rubber tubing. A vacuum was drawn up to the pressure plate and the stopcock turned to a closed position. A metal slug was used to rupture the glass pressure plate. In this way each sample gas could be introduced into the CEC Model 21-130 mass spectrometer. The remaining gas of each test specimen was then introduced into an evacuated 10 cm (3.9 in) gas cell for an infrared spectral analysis on the Perkin Elmer Model 21 infrared spectrophotometer.

The solid PPO foam specimens (including pre-test samples) were analyzed on the infrared spectrophotometer by a film technique. Benzene was determined to be a very suitable solvent for this material. Each specimen was dissolved in a small volume of the benzene solvent. The solution of the PPO foam was allowed to evaporate on a sodium chloride plate to yield a plastic film of suitable thickness for analysis.

As noted in Table 5-8 of test results, carbon dioxide was found to be a constituent of each specimen gas. There also was a noticeable absence of oxygen in each of the analyzed gas samples. Samples A, C, D, E₂ and F showed the presence of trichloroethane. The reason that the trichloroethane was not detected in E₁ and B may be attributed to the small sample quantity for test purposes and/or a less than optimum transferral of the gas specimen to the infrared gas cell. The trichloroethane is a blowing agent for the production of the foam. It also is called Chlorothene Nu under a Dow Chemical trade name. The trace quantities of Mass 35, 36 in the mass spectrometric analysis may be attributed to molecular fragments of the trichloroethane. All samples showed about the same weight loss except for the high density sample and the vacuum sample.

The PPO foam evolved trichloroethane which was detected in the gas atmospheres at the conclusion of the tests. This chemical compound is the principal blowing agent in the production of the PPO foam. The trichloroethane vapor probably was trapped in the cells of the PPO foam from the initial commercial production. The vapor was possibly released by cell rupture and/or diffusion during thermal soak.

Table 5-8. Analysis of Gas in Sample Tubes After Thermal Treatment

Specimen	Weight Change %	Mass Spectrometer Analysis							Infrared Analysis
		%	H ₂	H _e	N ₂	CO ₂	A _r	Other Trace	
A	-3.53	90.6			4.8	4.6		H ₂ O	Trichloroethane
B	-3.42			82.5	8.7	8.8		M35, 36	
C	-3.76				96.8	3.2			Trichloroethane
D	-2.79				99.7	0.3		M35, 36	Trichloroethane
E ₁	-4.12				50.7	49.3			
E ₂	-				49.3	50.7		M35, 36	Trichloroethane
F	-3.43				88.9	10.1	1.0		Trichloroethane

All of the samples of the gas atmospheres in which the PPO was thermally soaked showed the presence of carbon dioxide. Oxygen was noticeably absent in the samples. This indicates that an oxidation mechanism of some type is operating during the thermal soak.

Analysis of the foam itself by infrared spectral tests yielded data on the chemical composition of the material. Both the low density and high density materials did not show any changes when the data obtained after thermal soak was compared with data of the material prior to test. There was however a slight difference in data between the low density and high density material.

During the course of the infrared structural analyses of the PPO foam some chemical observations were made of the solubility of the foam. Benzene was used as a solvent to prepare the foam for infrared spectral analysis. All of the low density samples subjected to thermal soak showed a decreased solubility compared to the original material. The high density sample did not show this solubility alteration. A possible explanation of the solubility changes may be the fact that molecular cross linking and chain lengthening may be occurring. The increased molecular weight could result in decreased solubility.

Examination of the color photographs taken during the test and comparison of the test specimens with pre-test samples reveals that specimens A, B, C, and E all darkened slightly to the same degree while specimens D (high density foam in N_2) and F (air environment) darkened quite noticeably. When compared to the pre-test samples, the low density test specimens have a shriveled appearance.

Microscopic examination of the test specimens reveals a breakdown of the foam cell structure in the low density specimens. As a result of the thermal soak, the cell walls tend to break apart, opening passages for lateral gas convection and reducing foam strength. The microphotographs in Figures 5-2 and 5-3 reveal the pre-test and post-test low density foam structure. The pre-test photograph (Figure 5-2) shows several small voids (largest approximately 0.76 mm (0.03 inch)), but the openings in the cell walls are primarily due to the cutting knife. In the post-test photograph (Figure 5-3) three stages of cell wall breakdown can be seen. First the wall membrane acquires a frosted appearance. Then the wall tears apart leaving a ragged edge. Finally the wall opens completely. That the high density foam did not exhibit this cell wall breakdown is probably due to its much thicker cell walls.

Figures 5-4 and 5-5 are pre-test and post-test microphotographs of the high density foam. The dark spots in the foam are bubbles in the cell walls. Close examination of the pre-test samples (Figures 5-2 and 5-4) reveals many microscopic bubbles in the cell walls. In the low density foam, the bubbles are colorless with the exception of an occasional dark brown bubble. In the high density foam, many of the bubbles are dark brown with an occasional red bubble. After thermal soak, the bubbles in the low

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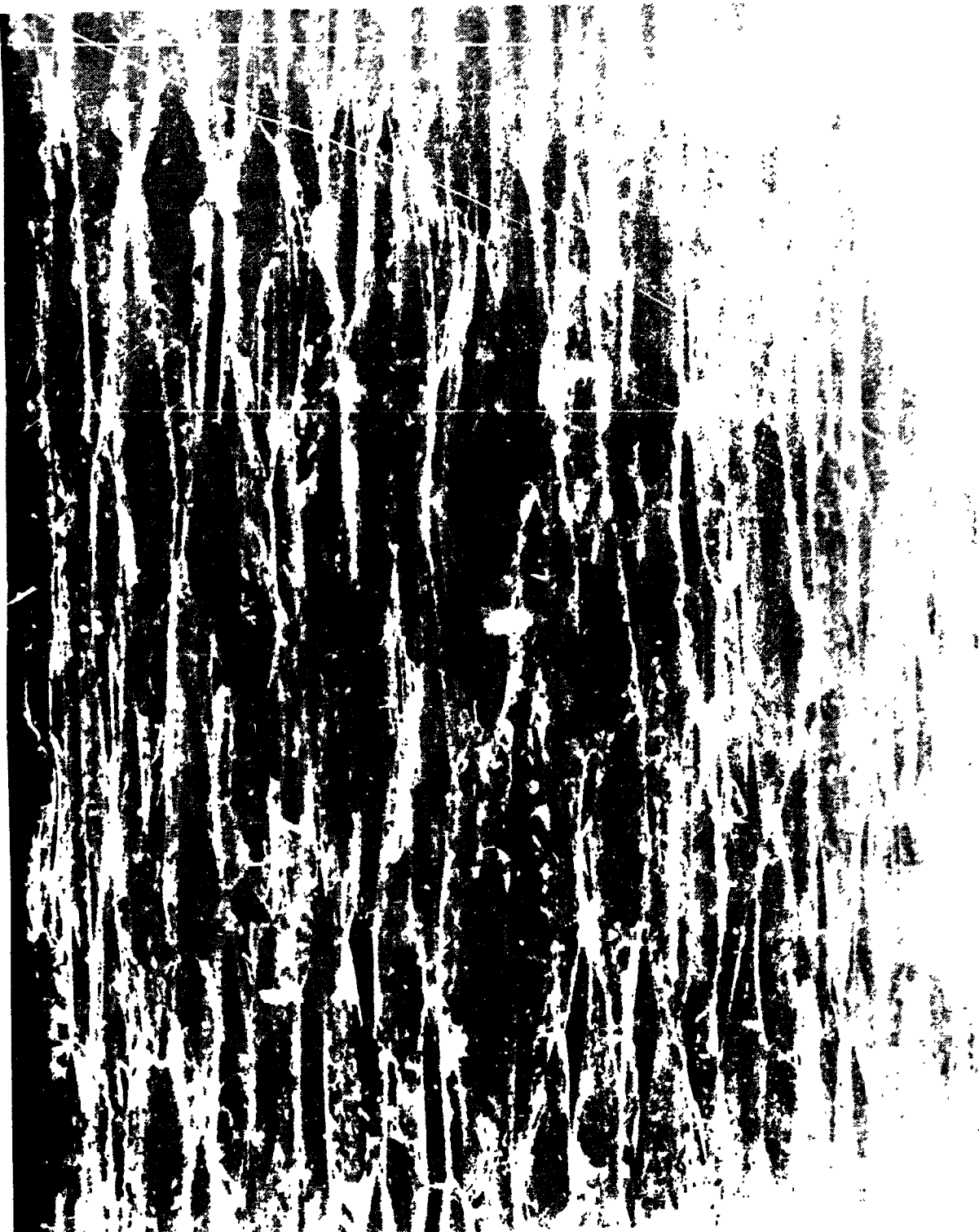


Figure 5-2. Pretest PPO Foam Sample, $20 - 30 \text{ kg/m}^3$ (1.87 pcf)

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Figure 5-3. Post Test PPO Foam Specimen, $20 - 30 \text{ kg/m}^3$ (1.87 pcf)

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Figure 5-4. Pretest PPO Foam Sample, $20 \sim 50 \text{ kg/m}^3$ (3.12 pcf)

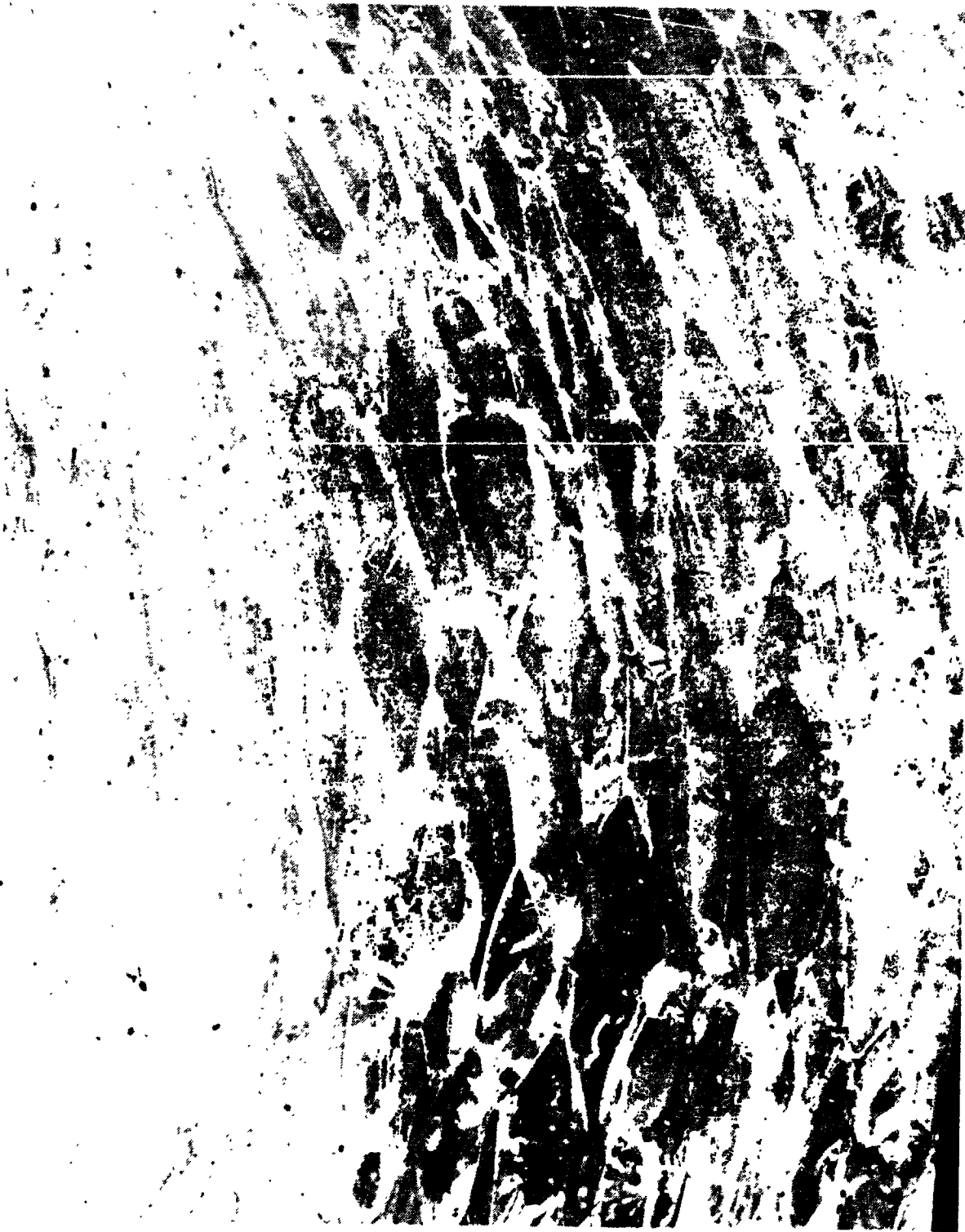


Figure 5-5. Post Test PPO Foam Specimen, 20 , 50 kg m³ (3.12 pcf)

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Figure 5-6. Post Test PPO Foam Specimen Cell Wall, 93 ., 50 kg/m³ (3.12 pcf)

density foam appear unchanged (Figure 5-3) while many of the bubbles in the high density foam turned dark brown (Figure 5-5). Figure 5-6 gives a close view of the cell wall of the post-test high density foam clearly showing the many clear and dark brown bubbles. The gasses entrapped in the bubbles are probably the blowing agent (DCE/CNU) and air. The dark brown color of some of the bubbles is possibly due to oxidation of the bubble wall by air entrapped in the bubble. The red material seen in a few of the bubbles is probably the Genitron blowing agent.

5.2 THERMAL CYCLING

Samples of PPO foam have been subjected to 100 temperature cycles from 21 to 450K (-422 to 350F) simulating the Space Shuttle life cycle. A cutaway drawing of the apparatus used for this test is shown in Figure 5-7. The nine 2.54 cm (1.00 in.) diameter by 2.54 cm (1.00 in.) thick PPO foam specimens were held in a copper can suspended from the positioning rod. The positioning rod, which passes through a seal at the top of the chimney, allows the specimen can to be held in the LH₂ bath, the heater or the lower section of the chimney. The chimney insulates the electric heater from the LH₂ bath by means of gas stratification. Thermocouples were located on the heater, in the center of the stack of foam specimens and at the outer surface of the specimens (see Figure 5-7). The cryostat insulates the LH₂ bath by means of a vacuum jacket, an LN₂ guard and external insulation. The foam specimens were placed in the specimen can, evacuated and helium backfilled, and maintained in an O₂ free atmosphere prior to placement into the cycling apparatus. A view of the thermal cycling test setup is shown in Figure 5-8.

A typical temperature cycle is shown in Figure 5-9. The test procedure for the 100 temperature cycles is:

Procedure for Temperature Cycling of PPO Foam

1. Raise specimen can into heater.
2. Turn heat power on (52V).
3. When specimens are heated to 450K (+350F), turn heater off and lower specimen can to halfway position.
4. When specimen temperature has dropped to 339K (+150F), lower the specimen can into the LH₂.
5. After the specimen can has been in the LH₂ bath for five minutes, raise the specimen can to the halfway position.
6. When the specimen temperature reaches 200K (-100F), raise the specimen can into the heater.
7. Turn heater power on (52V).

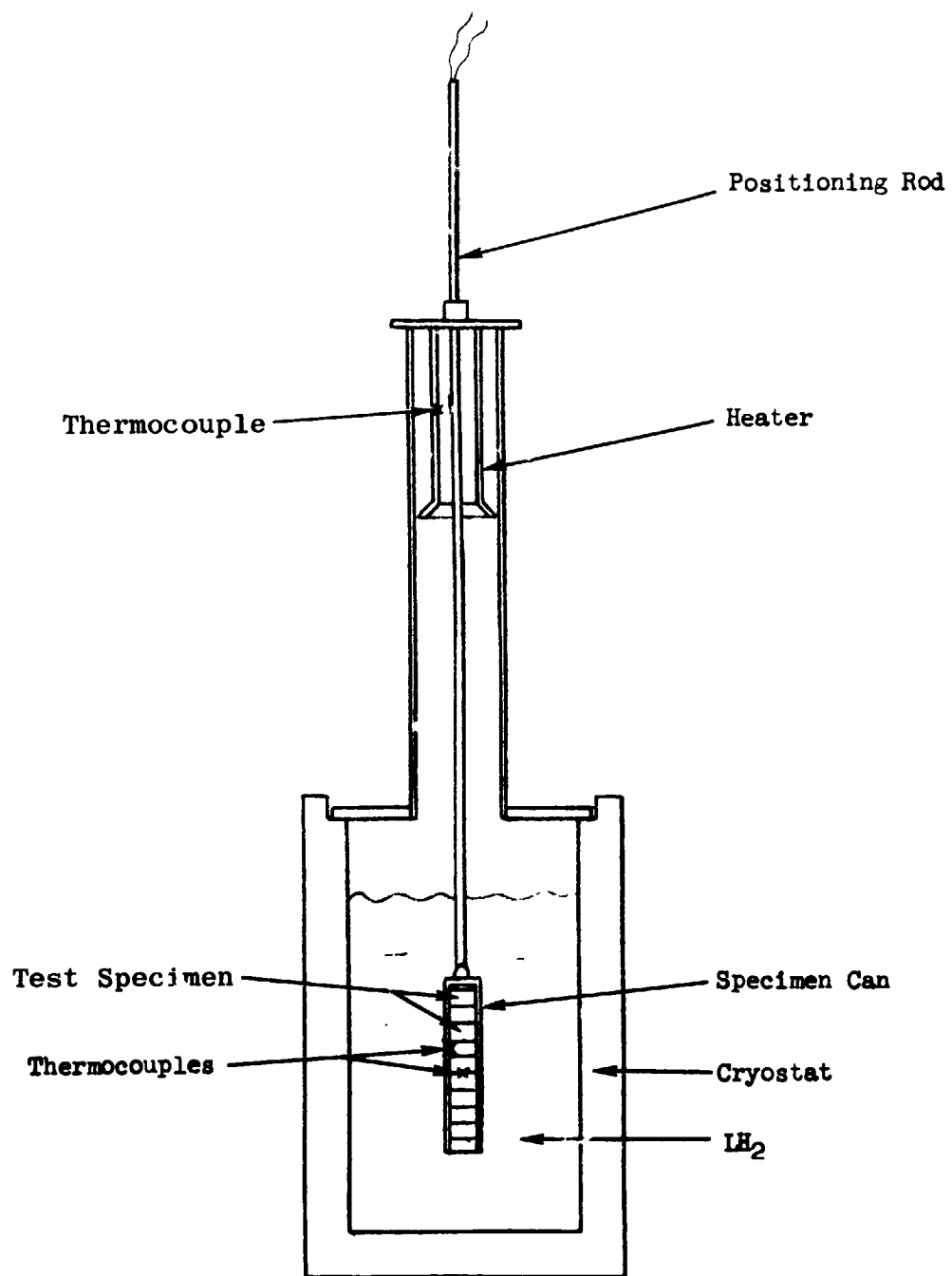


Figure 5-7. Thermal Cycling Apparatus

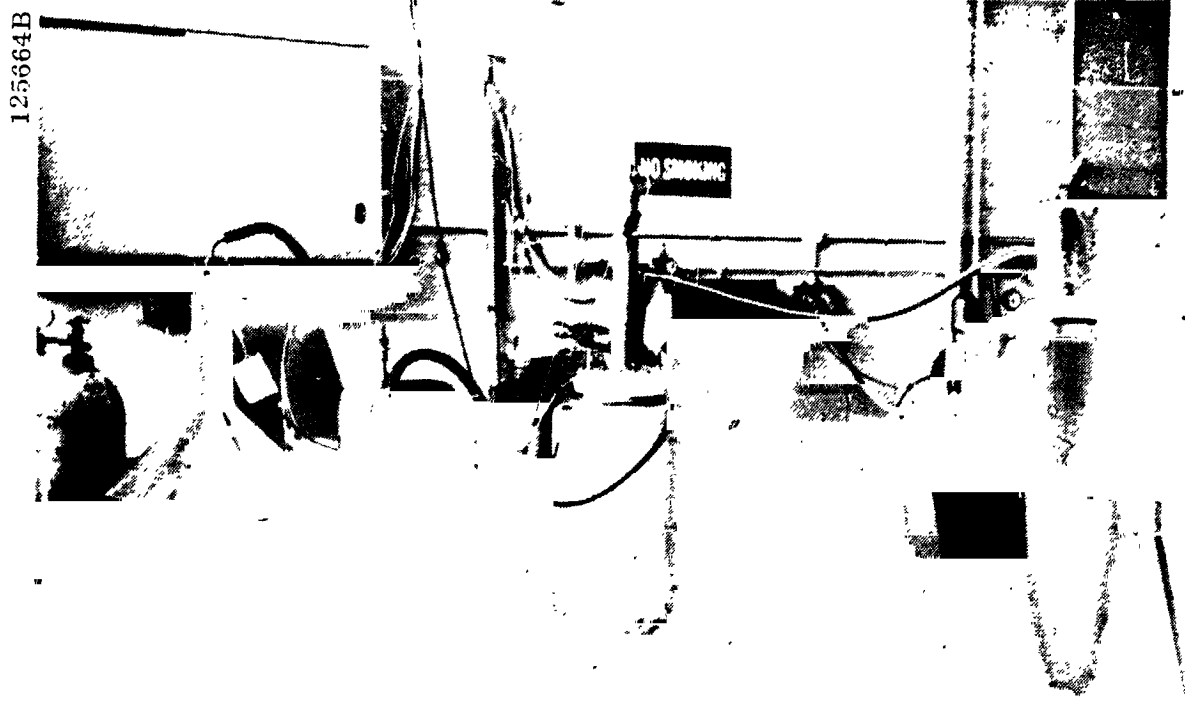


Figure 5-8. PPO Foam Thermal Cycling Test Setup

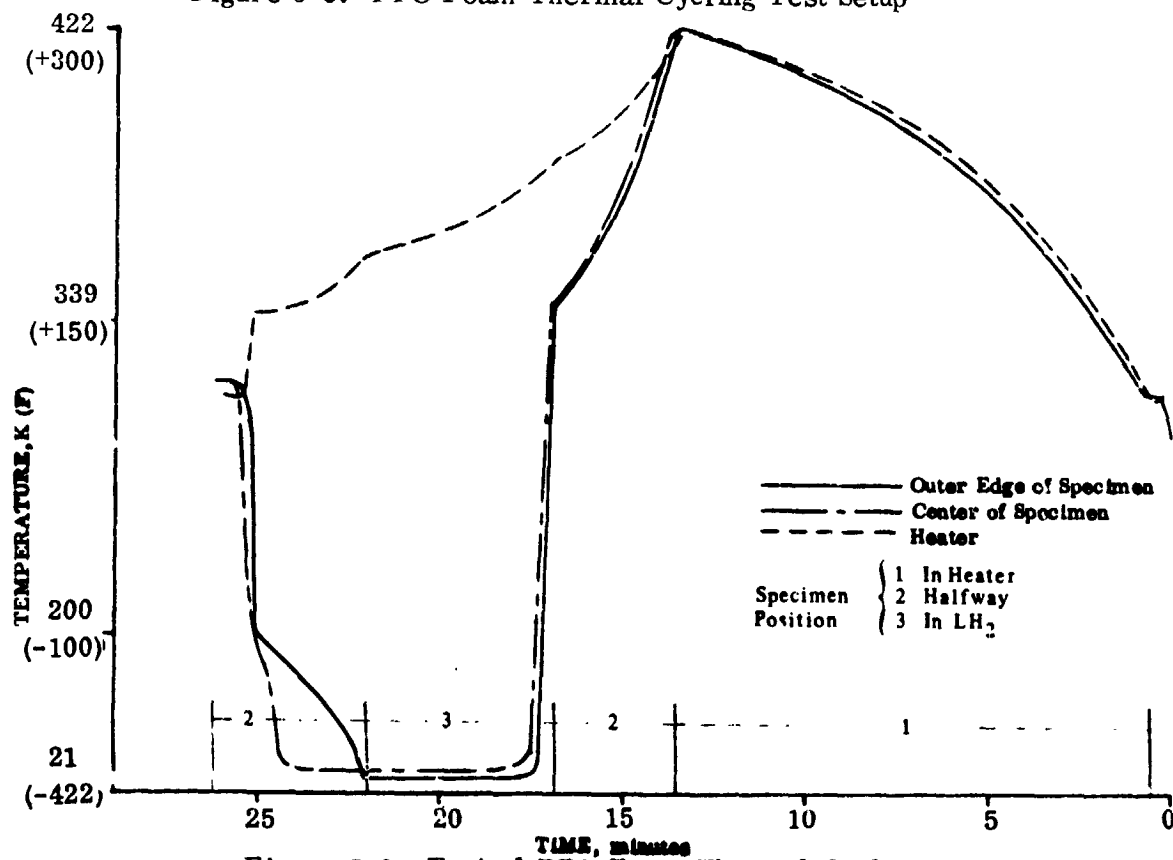


Figure 5-9. Typical PPO Foam Thermal Cycle

After completion of the 100 thermal cycles, the foam specimens were microscopically inspected for evidence of thermal aging. The only signs of thermal aging were the darkening of some of the bubbles in the cell walls and an ever-so-slight darkening in the general appearance of the foam. There was no visible change in cell structure. The weight of the specimens decreased by 3.7% and they shrank 1% in length (along the cells) and 0.4% in diameter (perpendicular to the cells). Longitudinal compressive strength tests were then carried out in the nine PPO foam specimens; three at 21K (-422F), three at 294K (70F) and three at 422K (300F). The results of these strength tests are shown in Figure 5-10 along with similar results obtained using virgin foam. All of the foam specimens, including the virgin material, were taken from the same PPO foam panel. Comparison of the strength data for the virgin and thermally cycled specimens shows no evidence of degradation within the normal scatter of the strength data.

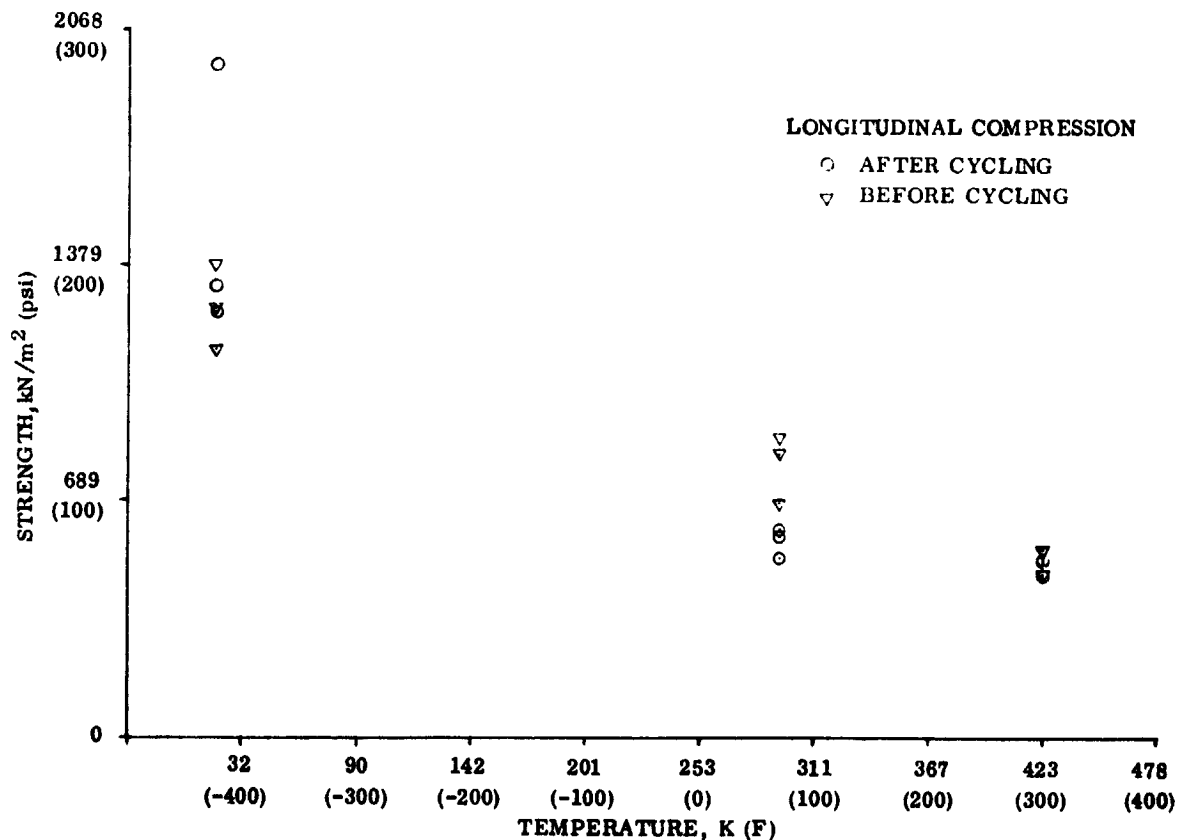


Figure 5-10. PPO Foam Thermal Cycling Strength Test Results

SECTION 6

THERMAL CONDUCTIVITY TESTS

A total of 17 guarded flatplate calorimeter tests were performed to determine the thermal conductivity of PPO foam in liquid hydrogen. The first eight tests were performed on 50 mm (1.97 in.) panels to screen various foam formulations prior to the selection of a preferred combination of blowing and nucleating agents for subsequent detailed configuration evaluation. These are referred to as "formulation screening" articles in Table 6-1. A preferred formulation was selected and 84 new panels, including 12 quality control panels destructively tested by the vendor, were manufactured in various thicknesses and densities. Eight foam configurations were selected from this group and tested thermally. They are listed under the heading "configuration screening" in Table 6-1. A final production test was run on a panel produced by hand but under carefully controlled conditions indicative of automated quality control; the last item in Table 6-1. The test specimen, facility, procedure and results are discussed below.

The performance of an open-cell insulation system is sensitive to more parameters than is a conventional closed-cell system. The open-cell insulation concept relies upon a balance between ullage pressure, liquid head, surface tension, and vapor pressure forces plus gas expansion due to heat transfer to provide an insulating gas layer between the cryogenic liquid and the warm tank wall. The LH_2 tank internal pressure and the orientation of the foam cells with relation to the gravity (or acceleration) vector, as well as the external thermal environment, affect the total heat flow through the insulation system. Consequently the test program evaluated the effects of cell orientation, source temperature, and tank pressure to fully characterize the thermal performance of the material.

The guarded flatplate thermal conductivity test specimen consists of two 33 cm (13 in.) diameter pieces of PPO foam with 0.64 mm (0.025 in.) deep by 27.9 cm (11.0 in.) diameter concentrically located area cut out on the inner face of both pieces. This recessed area is provided for the heater which is bonded between the two pieces of foam. The heater is composed of a guard section and a test section which are independently energized. The guard section minimizes the radial heat losses from the test section, providing a minimum temperature difference between the two sections. Thermocouples are located on the test and guard heaters to measure the ΔT during testing while other thermocouples measure temperatures in the insulation (Figure 6-1). To prevent liquid from penetrating into the insulation around the edges, the specimen is wrapped with 181 style fiberglass cloth and coated with a polyurethane

Table 6-1. Thermal Conductivity Test Pieces

Panel Number	Blowing Agent	Nucleating Agent Material	Quantity pph ⁽¹⁾	Panel Nominal Thickness ⁽²⁾ mm (in.)	Test Pieces Thickness ⁽³⁾ mm (in.)	Density kg/m ³ (lb/ft ³)
A. Formulation Screening						
71-23	DCE/CNU ⁽⁴⁾ 1:3	Ver ⁽⁵⁾	2	50 (1.97)	44.7 (1.76)	45.3 (2.83)
71-19	DCE/CNU 1:1	"	"	"	"	43.4 (2.71)
71-24	DCE/CNU 1:1	"	"	"	45.0 (1.77)	44.5 (2.78)
71-21	DCE/CNU 3:1	"	"	"	45.2 (1.78)	46.8 (2.92)
71-30	DCE/SBP 5:1	"	5	"	"	47.4 (2.96)
71-31	DCE/SBP 5:1	Gen	2	"	"	37.8 (2.36)
71-33	DCE	"	"	"	"	46.8 (2.92)
71-35	DCE	Ver	5	"	45.0 (1.77)	43.2 (2.70)
B. Configuration Screening						
72-74	DCE/CNU 1:3	Gen	2	75 (2.95)	24.6 (0.97)	61.5 (3.84)
72-41	"	"	"	"	24.9 (0.98)	38.0 (2.37)
72-1	"	"	"	"	24.4 (0.96)	28.8 (1.80)
72-64	"	"	"	25 (0.98)	24.9 (0.98)	52.4 (3.27)
72-56	"	"	"	"	25.4 (1.00)	41.5 (2.59)
72-17	"	"	"	"	24.9 (0.98)	28.5 (1.78)
72-30	"	"	"	50 (1.97)	46.0 (1.81)	42.1 (2.63)
72-32	"	"	"	"	14.5 (0.57)	40.5 (2.53)
C. Final Production Test						
75-10	"	"	"	50 (1.97)	45.2 (1.78)	41.0 (2.56)

(1) Parts per hundred parts of resin

(2) Specified thickness

(3) Two pieces required per test specimen

(4) DCE - dichloroethane, CNU - Chlorothene Nu (trichloroethane), SBP - petroleum ether

(5) VER - vermiculite, GEN - Genitron (azodicarbonamide)

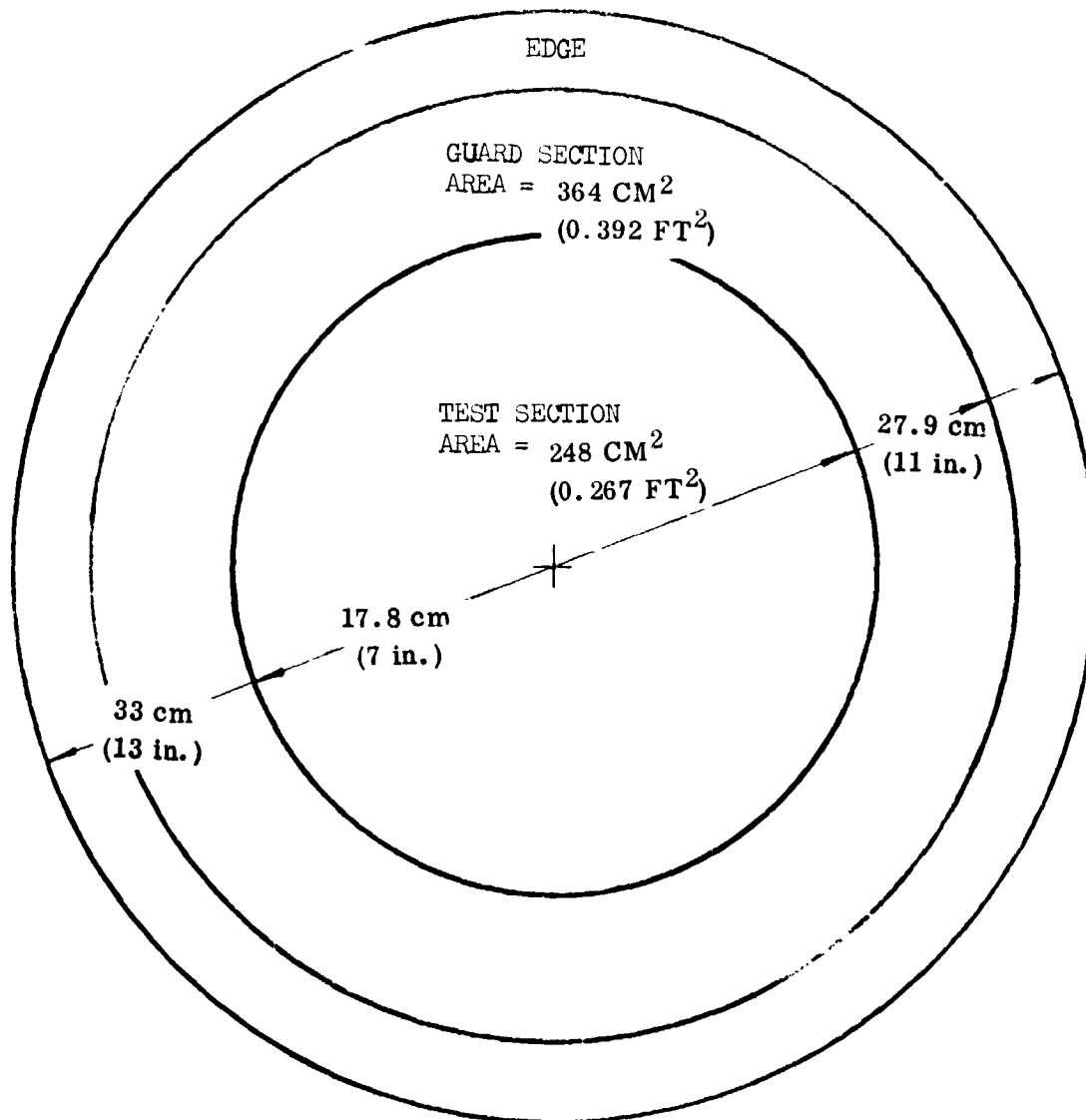
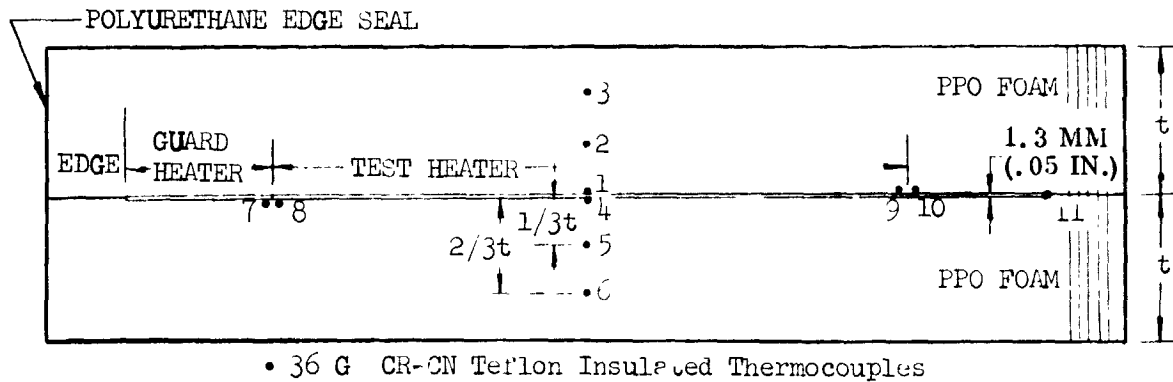


Figure 6-1. PPO Foam Thermal Conductivity Apparatus

sealant. The first specimen used only sealant but the sealant became brittle and cracked when exposed to the LH_2 (Figure 6-2). Therefore only a thin coating of sealant is used with the cloth. Since reinforcing cloth has been used, no failures resulted. A photograph of a fiberglass-wrapped specimen is shown in Figure 6-3.

A thermal analysis of the PPO foam thermal conductivity test specimen was made utilizing a steady state version of Convair's 2162 computer program. The analysis was performed to evaluate the radial heat flow within the specimen. The radial heat flow constitutes an error in determining the thermal conductivity of the specimen. Figure 6-4 depicts the model used for the analysis. The slash lines indicate an adiabatic wall. The "Z" dimension varies along the "X" axis because the model is pie shaped. No heat transfer is assumed to occur in the "Z" direction. Other boundary conditions include a constant heat flux value for guard heater nodes 71 through 76 and a constant heat flux value for node 90, a test heater node (the value may or may not coincide with that of nodes 71 through 76). Also, the nodes around the periphery are flagged as heat sinks corresponding to the fluid temperature of 22K (40R).

The temperature change in the radial direction must be kept as small as possible. This can be accomplished by regulating individually the power in the test and guard sections. This was done with the computer model, holding the guard heater power constant and varying the test section power until the ΔT between nodes 71 and 90 was essentially zero. Figure 6-5 depicts the results of varying the test section heater power. The results were converted to a curve for determining the percent radial heat loss $[(\text{radial heat loss}/\text{total test section power input}) \times 100]$ as a function of the temperature gradient at the interface of the test section and the guard section heaters (Figure 6-6). Figure 6-7 illustrates the steady state temperature distribution which results from imposing a guard section heat flux of 2664 W/m^2 (845 Btu/hr-ft^2) and a test section heat flux of 2050 W/m^2 (650 Btu/hr-ft^2).

Data from the first PPO foam thermal conductivity tests indicated large temperature gradients on the hot face of the test specimen. Temperature differences as great as 14K (25R) occurred between thermocouples No. 1 and No. 8 (Figure 6-1). It was suspected that the thin gage aluminum as well as the wide spacing of the heater wires, as much as 2.5 cm (1.0 in.) in the center, did not adequately distribute the heat. A thermal analysis of the heater section (Figure 6-8) was made in an effort to determine the causes and solutions to large temperature gradients on the hot face of the specimen. Figure 6-9 depicts the computer model used. The slash lines indicate an adiabatic wall. Nodes 1, 11, 21, 31, 41, 51, 61, 71, and 81 are held at LH_2 temperature 21K (38R) while node 10, representing the heater wire, is held at 333K (600R). The "Z" dimension is 1.27 mm (0.050 in.) while all other dimensions are as indicated. Steady state temperatures were computed for aluminum foil thicknesses of 0.076 mm and 0.127 mm (0.003 and 0.005 in.). The temperature gradients along the aluminum for the two cases are depicted in Figure 6-10. The maximum ΔT occurring in the 0.076 mm (0.003 in) aluminum is about 5.5K (10R). This value

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Figure 6-2. Cracked Polyurethane Edge Seal

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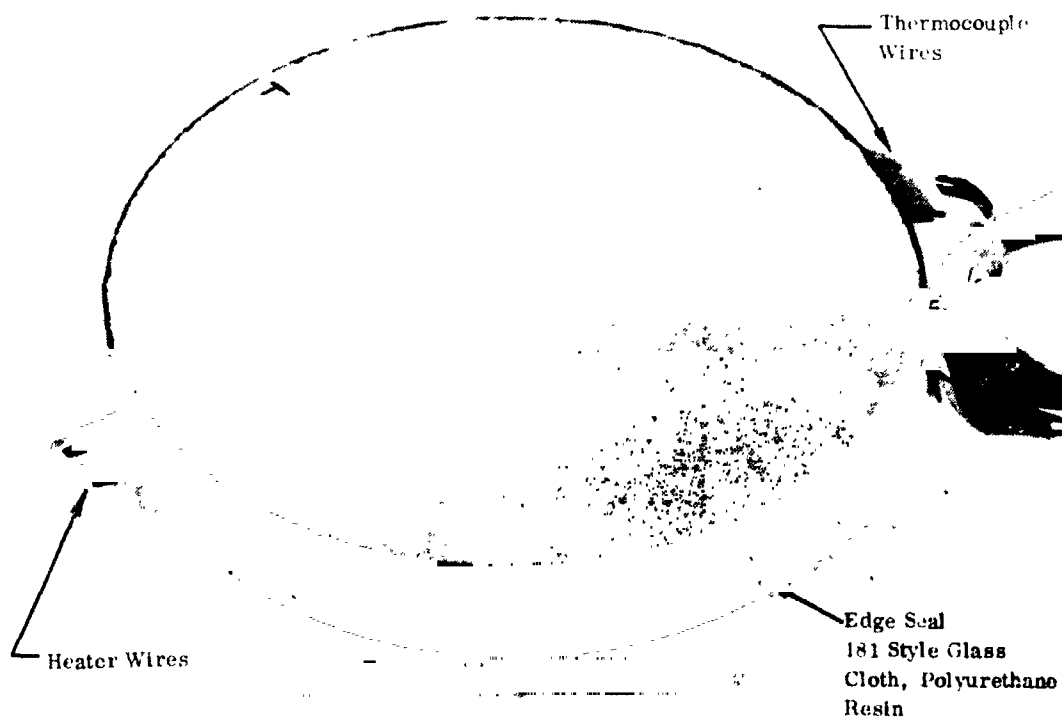


Figure 6-3. Thermal Conductivity Test Specimen

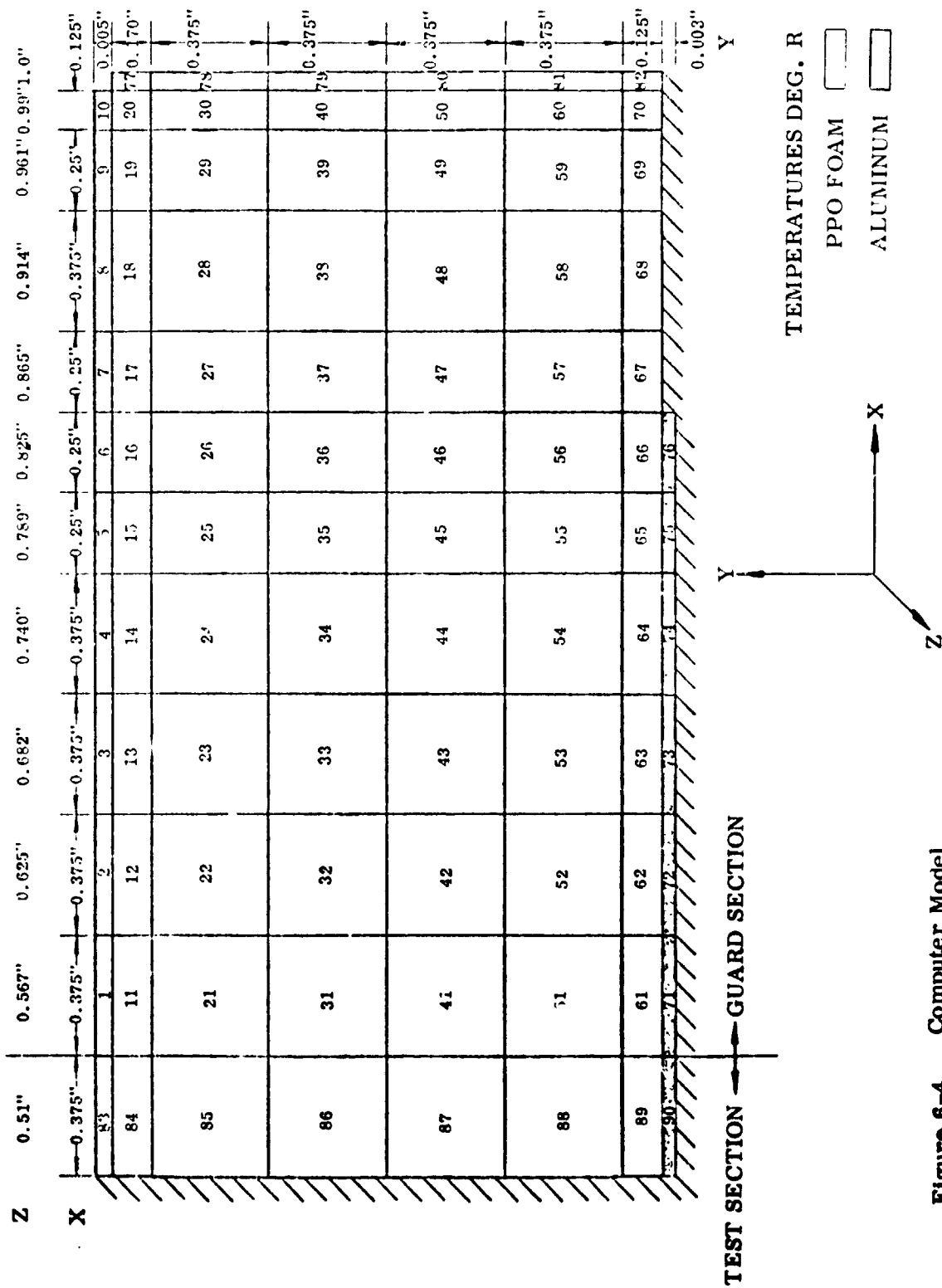


Figure 6-4. Computer Model.

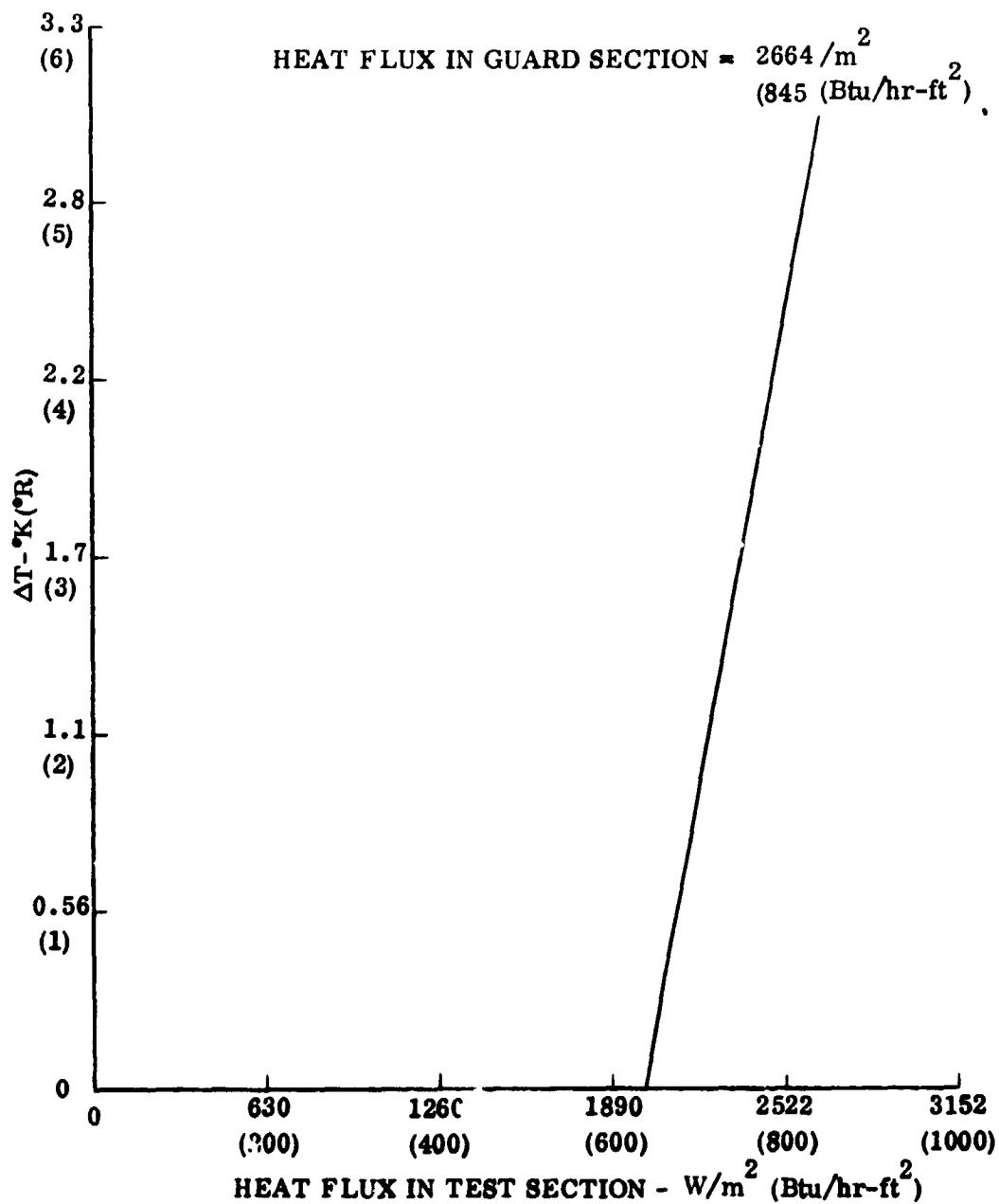


Figure 6-5. Computer Model Prediction of Temperature Difference Vs Test Section Heat Flux

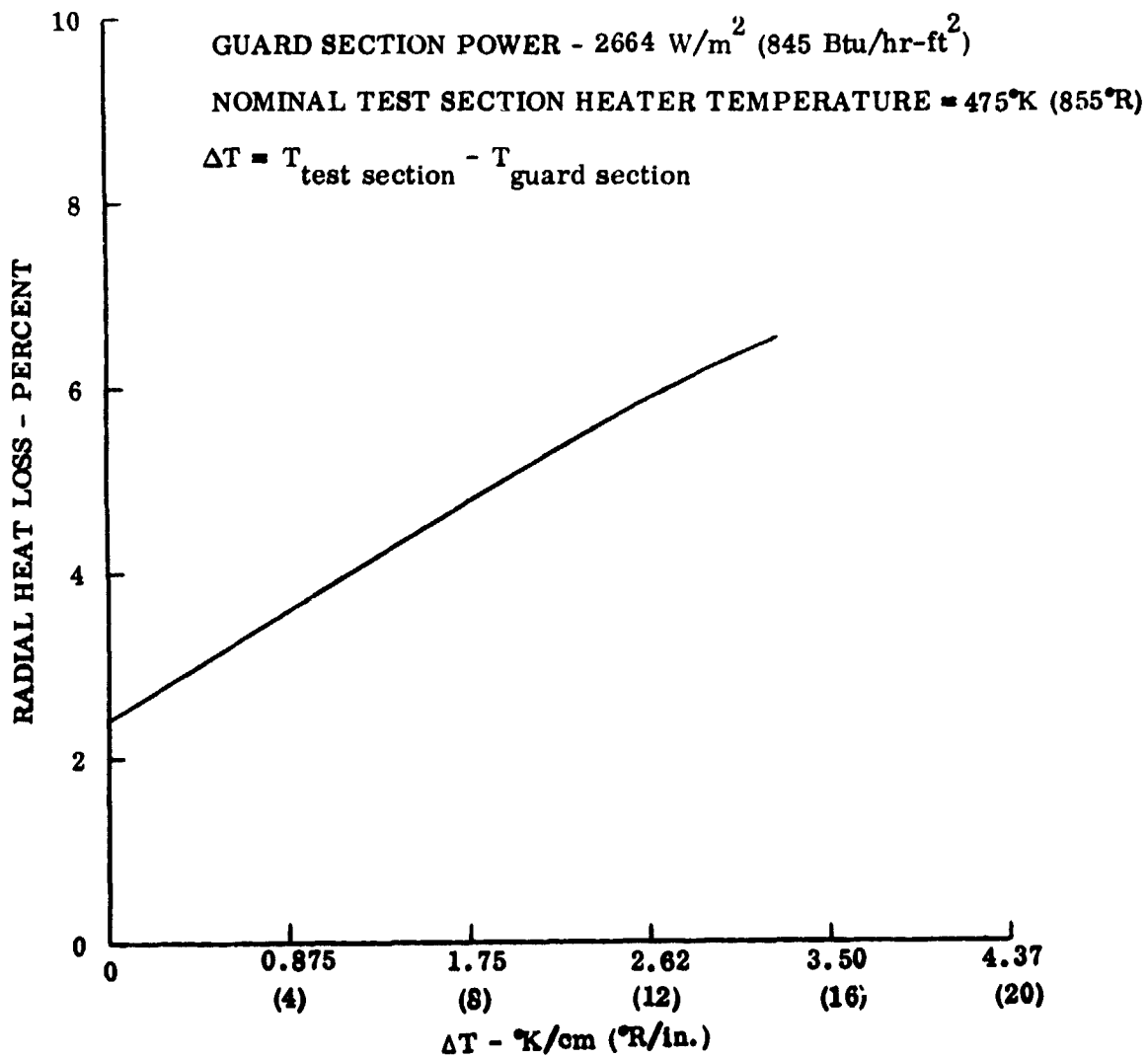


Figure 6-6. Radial Heat Loss from the Test Section as a Function of Temperature Gradient

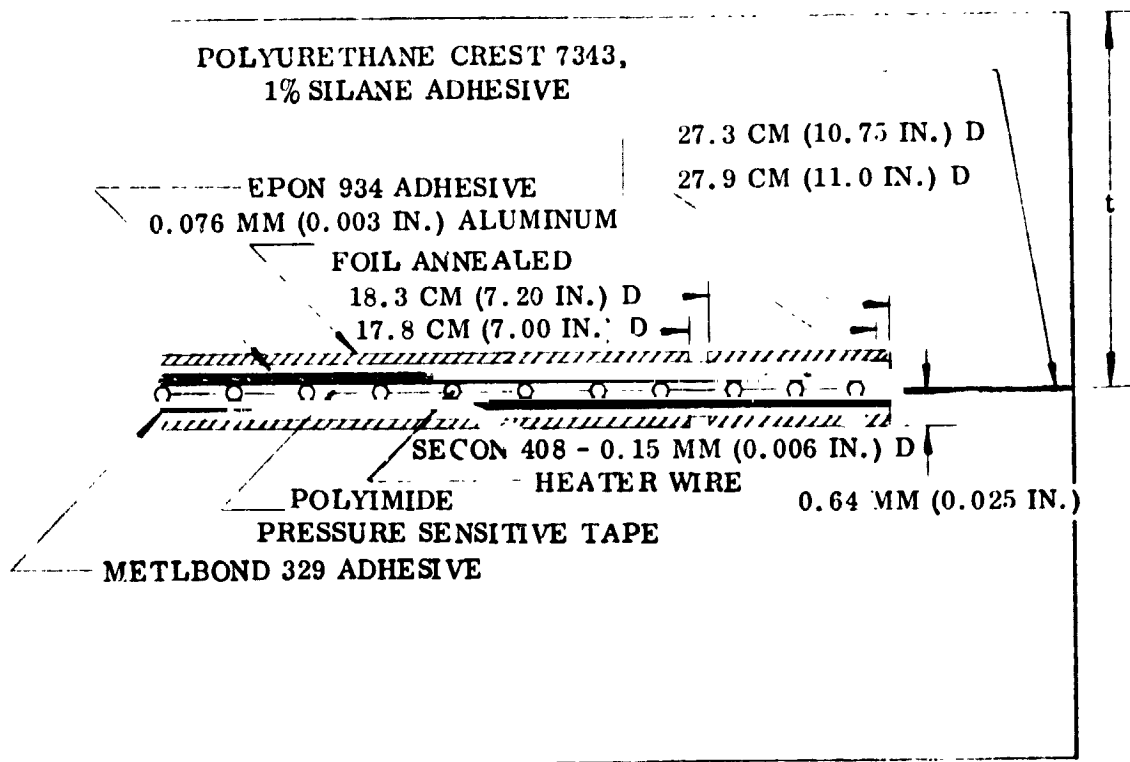


Figure 6-8. Heater Detail

compares quite well with the test results for the same temperature range. The 14K (25R) ΔT found in the test results occurred at a much higher temperature condition. Increasing the aluminum thickness to 0.127 mm (.005 in) reduces the maximum ΔT to 3.3K (6R). As a result of this investigation, a new heater was designed. Minco Products Inc. was selected for manufacture. The heating elements are spaced about 2.5 mm (0.1 in.) apart and are insulated with Kapton tape. The heater is bonded between two 0.127 mm (0.005 in.) thick sheets of aluminum foil.

All thermal conductivity tests were conducted at the Convair Sycamore Canyon LH₂ test site. The test specimen was immersed in LH₂ in a thermally guarded tank shown in Figure 6-11. The fixture for holding and orienting the test specimen is illustrated in Figure 6-12a for the horizontal orientation and Figure 6-12b for the vertical orientation. The specimen can be moved from horizontal to vertical during the test without opening the tank. Temperatures in the apparatus were monitored by 36 gauge Chromel-Constantan thermocouples in conjunction with a Dymec Data Acquisition System. There are a total of 11 T.C.'s located in the specimen, Figure 6-13. Whenever possible the wires were routed along isotherms to minimize large temperature gradients. T.C. 1 was used to control the test section source temperatures and T.C.'s 9 and 10 were used to monitor the temperature difference between the test and guard heaters. The guard heater power was adjusted to minimize this temperature difference. Separate power supplies were provided for

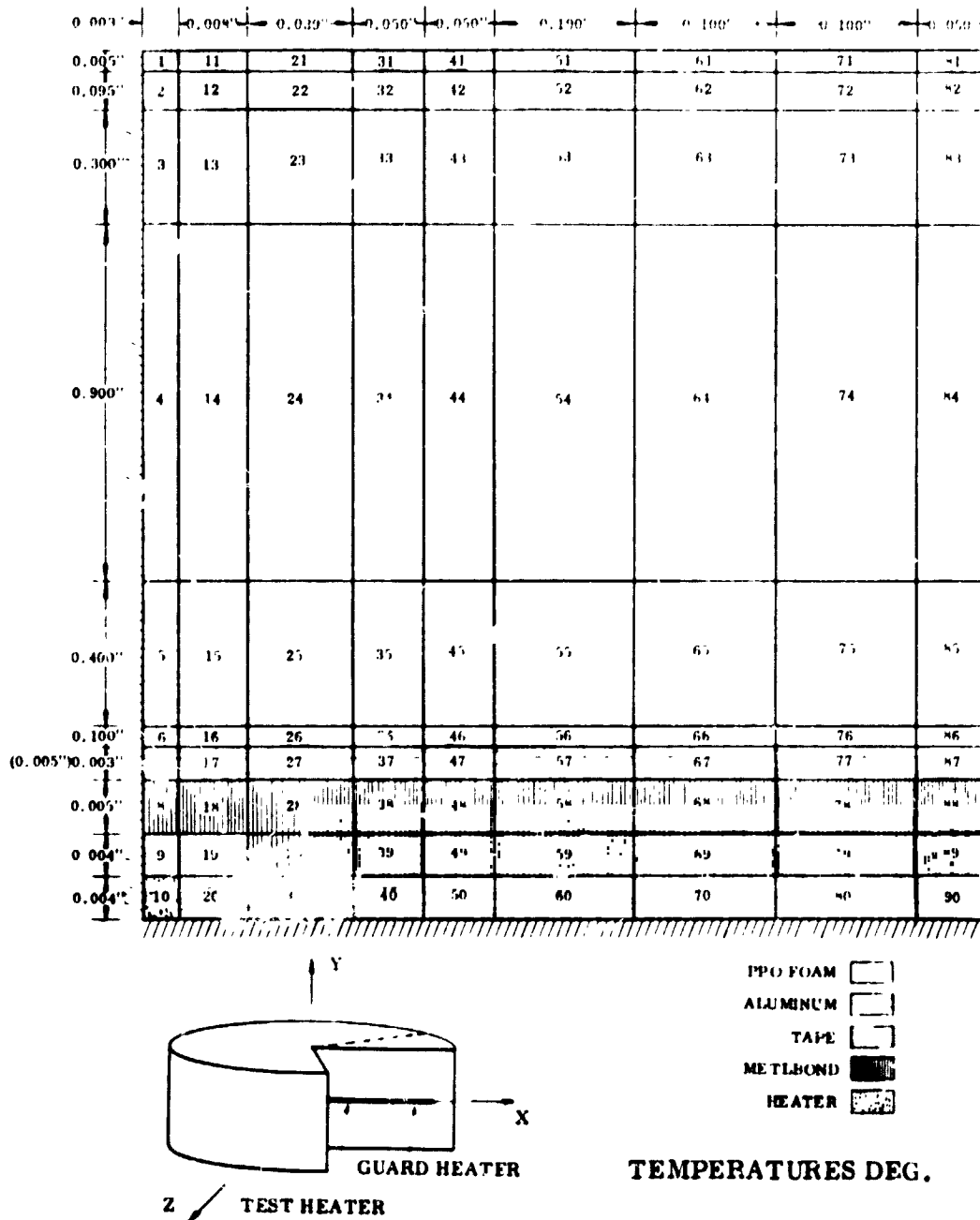


Figure 6-9. Computer Nodal Model for Heater Analysis

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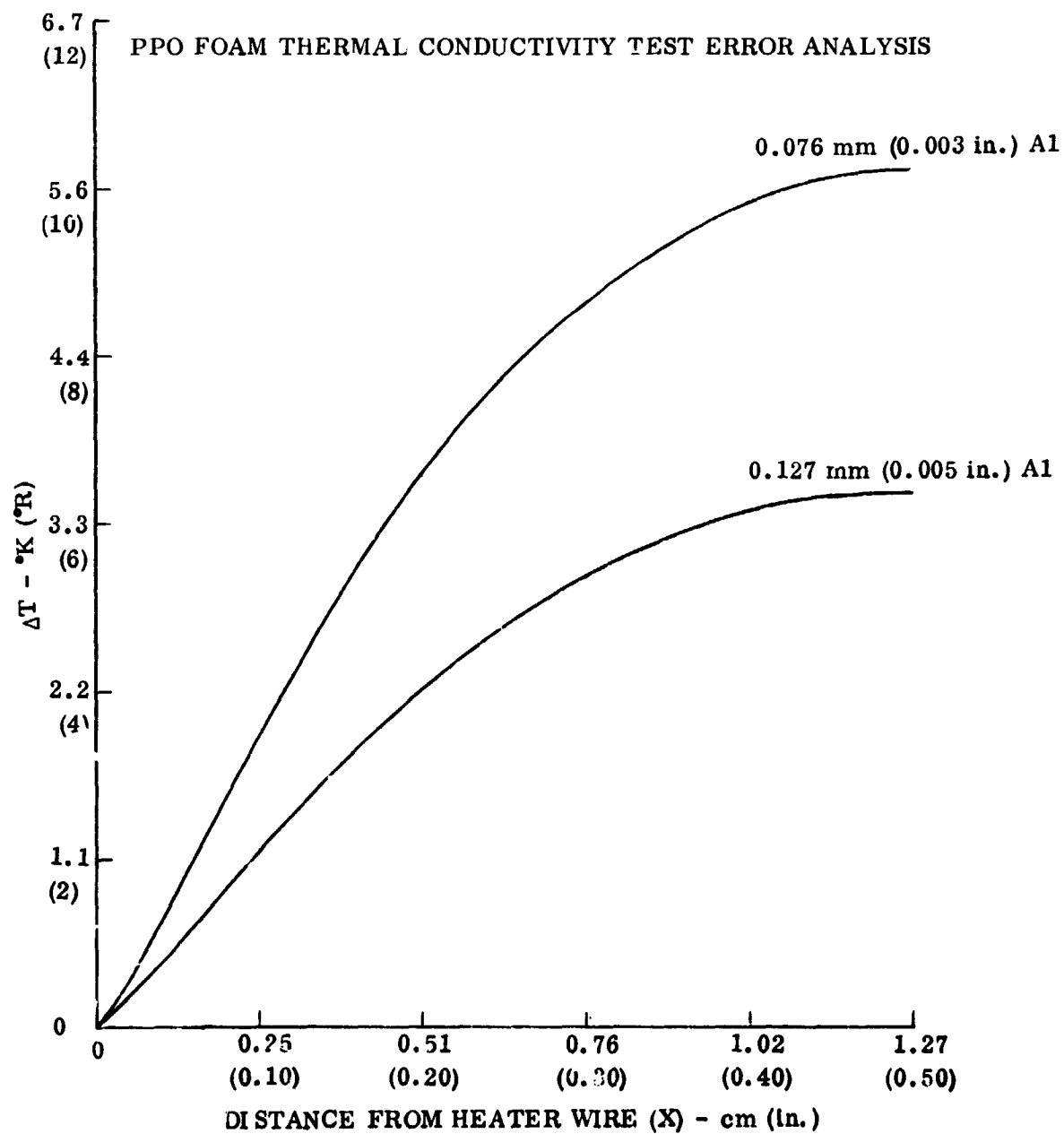


Figure 6-10. ΔT Distribution in Aluminum Heater Plate.

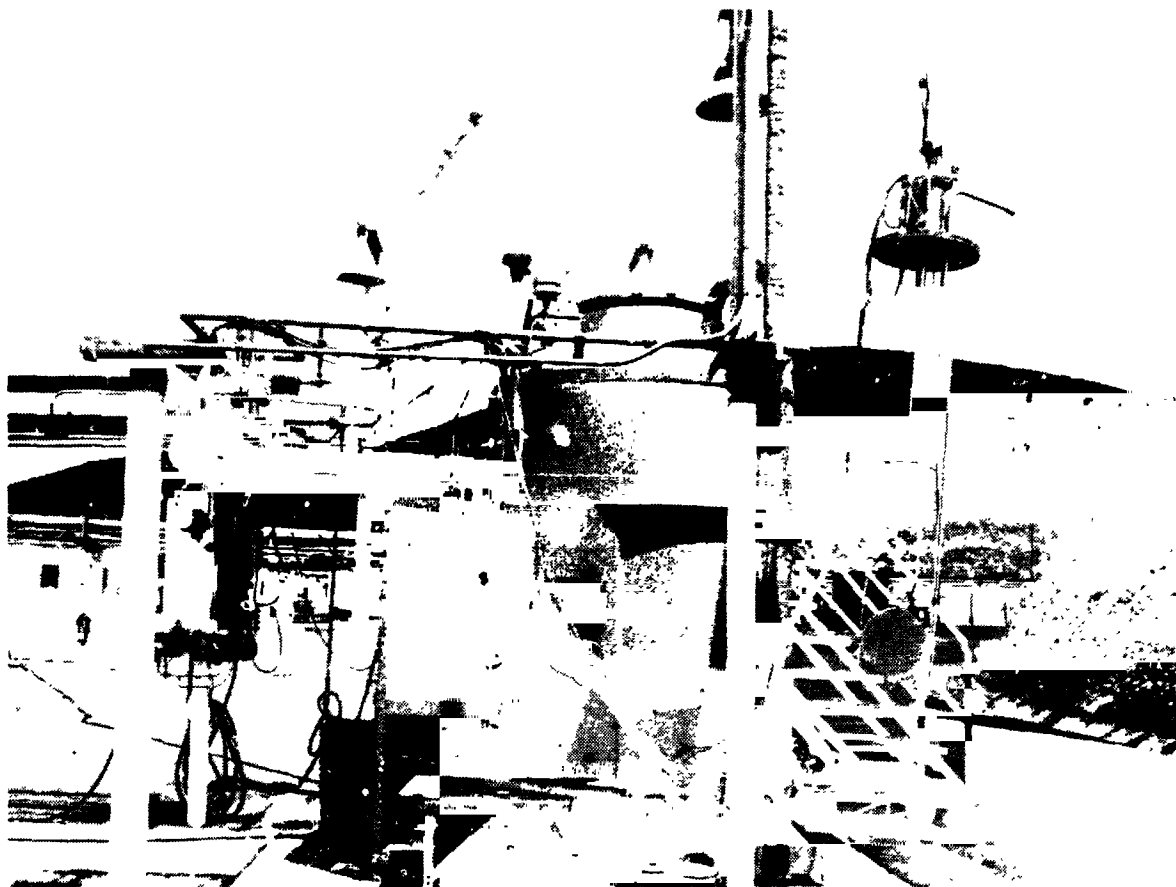


Figure 6-11. Thermal Conductivity Test Tank

the test and guard heaters. The supplies were adjusted manually, controlling the power to ± 3 percent. An absolute pressure transducer was used to measure the tank ullage pressure. All temperatures, voltages, amperages, and the tank pressure were measured, printed on tape, and punched on numerical tape by the Dymec system. The test data was reduced to engineering units by a Varian Data 620 computer and tabulated by a Stromberg-Carlson 4020 plotter. The detailed test procedure is presented in Appendix B.

An effective thermal conductivity of the insulation was calculated at each equilibrium data point. Since the two halves of the test specimen are the same thickness and the same material, it was assumed that the power generated in the test section heater was equally transferred through the two sections. Therefore the effective thermal conductivity of the foam/LH₂ system is computed using the Fourier equation where Q is the total power generated in the test section, t is the insulation thickness (one-half the specimen thickness), A is the test section area and ΔT is the temperature difference between T. C. 1 and the saturation boiling temperature of LH₂:

$$K_{\text{eff}} = \frac{t}{\Delta T} \frac{Q}{2A}$$

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Figure 6-12a. Thermal Conductivity Test Specimen in the Horizontal Orientation

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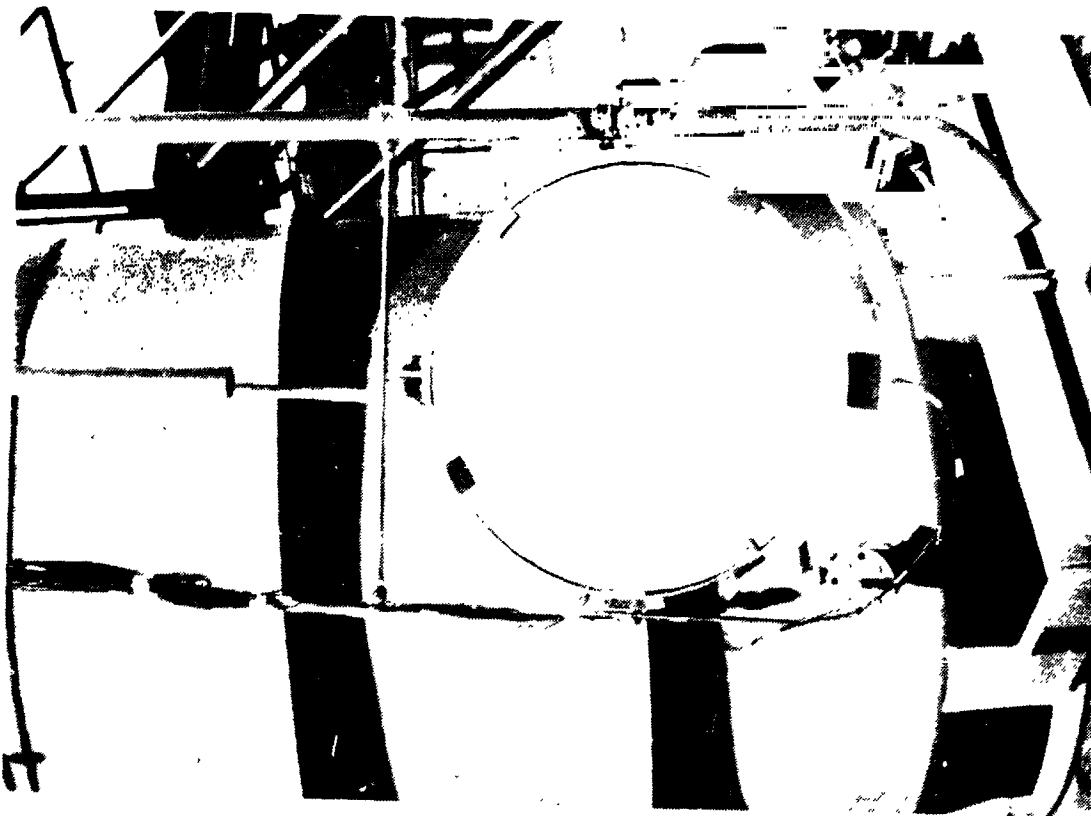


Figure 6-12b. Thermal Conductivity Test Specimen in the Vertical Orientation

Since the thermal conductivity of an internal, open cell, insulation system cannot be lower than the conductivity of the gas to which the insulation is exposed, the test results are presented in the form of a conductivity ratio of the foam system to hydrogen gas, where a ratio of 1.0 is minimum. The thermal conductivity of gaseous hydrogen is depicted in Figure 6-13. Equilibrium hydrogen in the liquid state is nearly 100 percent para therefore the thermal conductivity data for para hydrogen is used in the data reduction. The data has been curve-fitted and the equation is also presented in Figure 6-13. It should be noted that the equation is valid only between 22 and 244K (40 and 440R).

6.1 FORMULATION SCREENING

Thermal conductivity ratio data for four of the formulation screening panels plus three panels tested prior to contract initiation are presented in Figures 6-14 through 6-17. The two unnumbered panels were manufactured using the dichloroethane blowing agent and the vermiculite nucleating agent. The formulations for the remaining panels are described in the Appendix A.

All of the horizontal orientation (cells-vertical) data taken at a pressure of 107 kN/m^2 (15.5 psia) is presented in Figure 6-14. The conductivity ratios range from a low of 1.1 to a high of 2.1, with a trend towards higher conductivity ratios at lower mean temperature ($T_m = (T_{\text{hot}} + T_{\text{LH}_2})/2$). The 21.6 mm (0.85 in.) thick specimen was quite constant over a broad temperature range with the conductivity ratio remaining around 1.4. The poorest performer at low mean temperatures was the 68.6 mm (2.7 in) specimen while at increased temperature the performance was good. With the insulation in the horizontal orientation an increase in the tank pressure to as high as 462 kN/m^2 (67 psia) increased the conductivity ratio by only 12 percent (Figure 6-15). The 21.6 mm (0.85 in.) thick specimen was insensitive to pressure increase.

Test data taken with the specimen in the vertical orientation (cells-horizontal) is depicted in Figures 6-16 and 6-17. Again the 21.6 mm (0.85 in.) thick specimen was insensitive to temperature, pressure or orientation change. An examination of the 68.6 mm (2.7 in) specimen after testing revealed no apparent failures.

Four additional thermal conductivity tests were performed on formulation screening panels 71-30, 31, 33 and 35. The 33-cm (13 in.) diameter symmetrical test specimens were fabricated in the same manner as were the earlier specimens. However, several changes were made in the original thermal conductivity test procedure. During tests of panels 71-19, 21, 23 and 24 the tank ullage pressure was raised slowly from 107 kN/m^2 (15.5 psia) to 276 kN/m^2 (40 psia) to determine the effect of absolute pressure level on thermal conductivity. No change was made in the heater power setting and no significant changes occurred in any of the temperatures recorded in the specimens. Thus the thermal performance of high quality foam is independent of the absolute pressure level between these limits when the pressure is changed slowly. Consequently pressure change controls were eliminated. The revised test plan is presented in Appendix B.

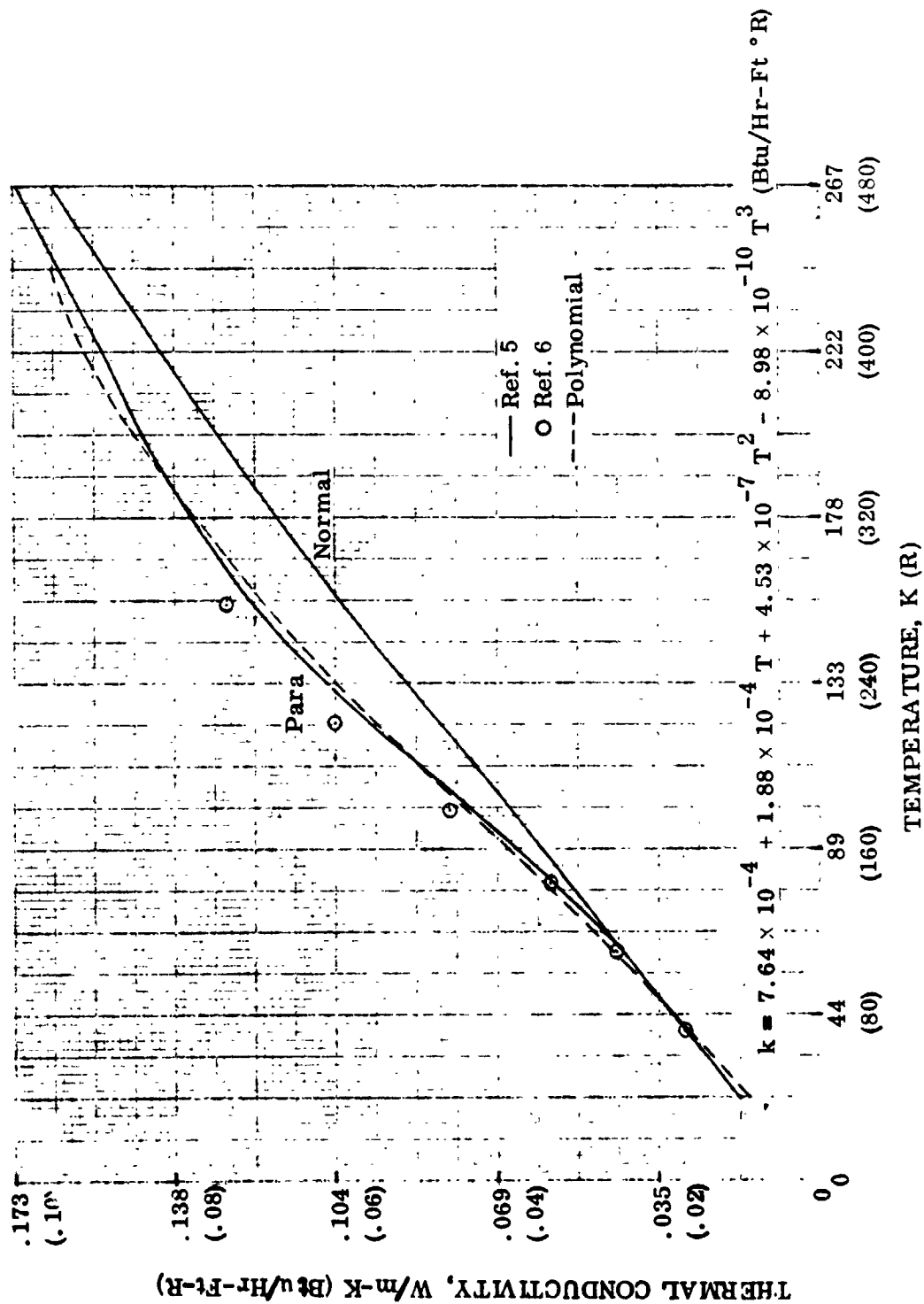


Figure 6-13. Thermal Conductivity of Gaseous Hydrogen at 1 Atm.

PANEL NO.	PANEL THICK. mm	PANEL THICK. (in.)	TEST THICK. mm	TEST THICK. (in.)	SYMBOL
-	51	(2)	45.7	(1.80)	○
-	51	(2)	45.7	(1.80)	●
71-3	76	(3)	21.6	(0.85)	□
71-4	76	(3)	68.6	(2.70)	■
71-23	51	(2)	45.7	(1.80)	△
71-21	"	"	"	"	▲
71-19	"	"	"	"	○
71-24	"	"	"	"	●

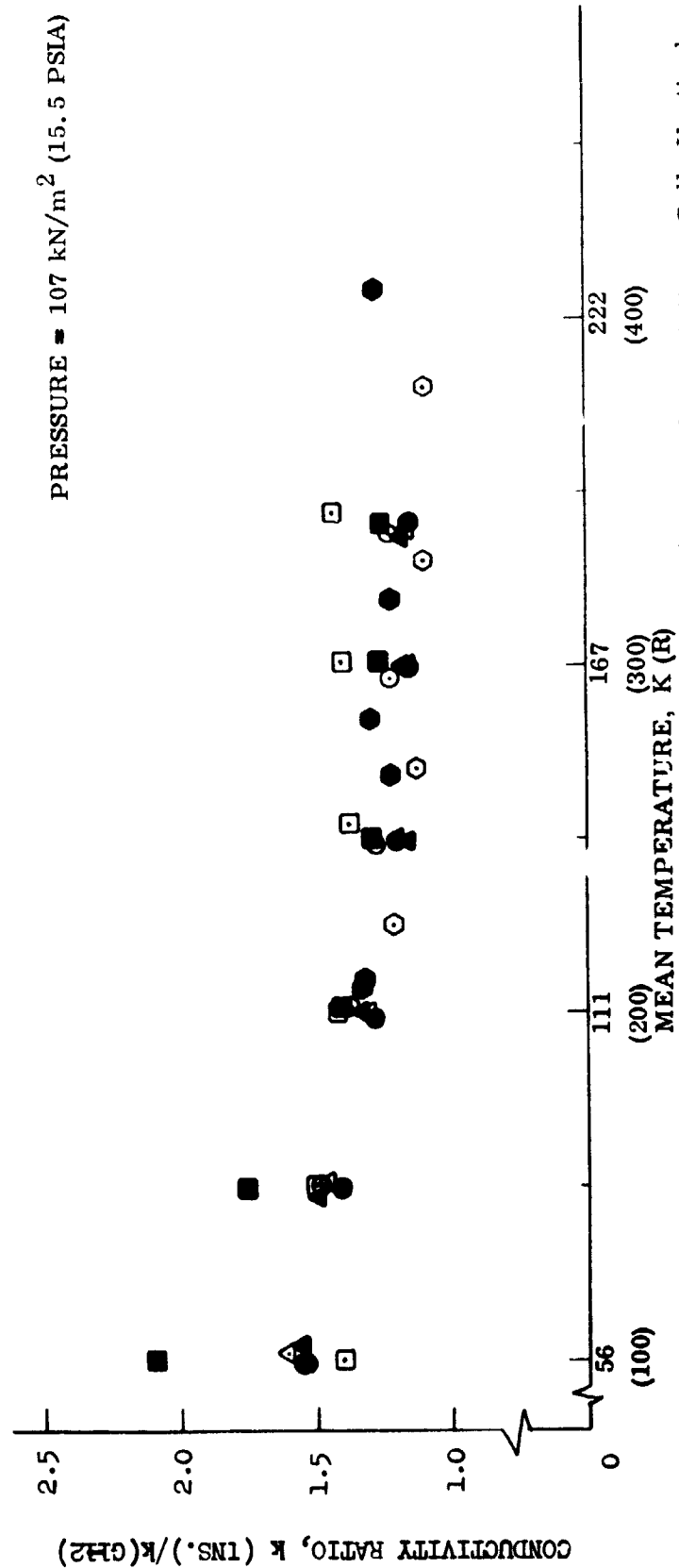


Figure 6-14. PPO Foam Thermal Conductivity in LH₂, Horizontal Orientation - Cells Vertical

PANEL NO.	PANEL THICK. mm (in.)	TEST THICK. mm (in.)	SYMBOL
-	51 (2)	45.7 (1.80)	●
71-3	76 (3)	21.6 (0.85)	□
71-4	76 (3)	68.6 (2.70)	■
71-23	51 (2)	45.7 (1.80)	△
71-21	" "	" "	▲
71-19	" "	" "	○
71-24	" "	" "	●

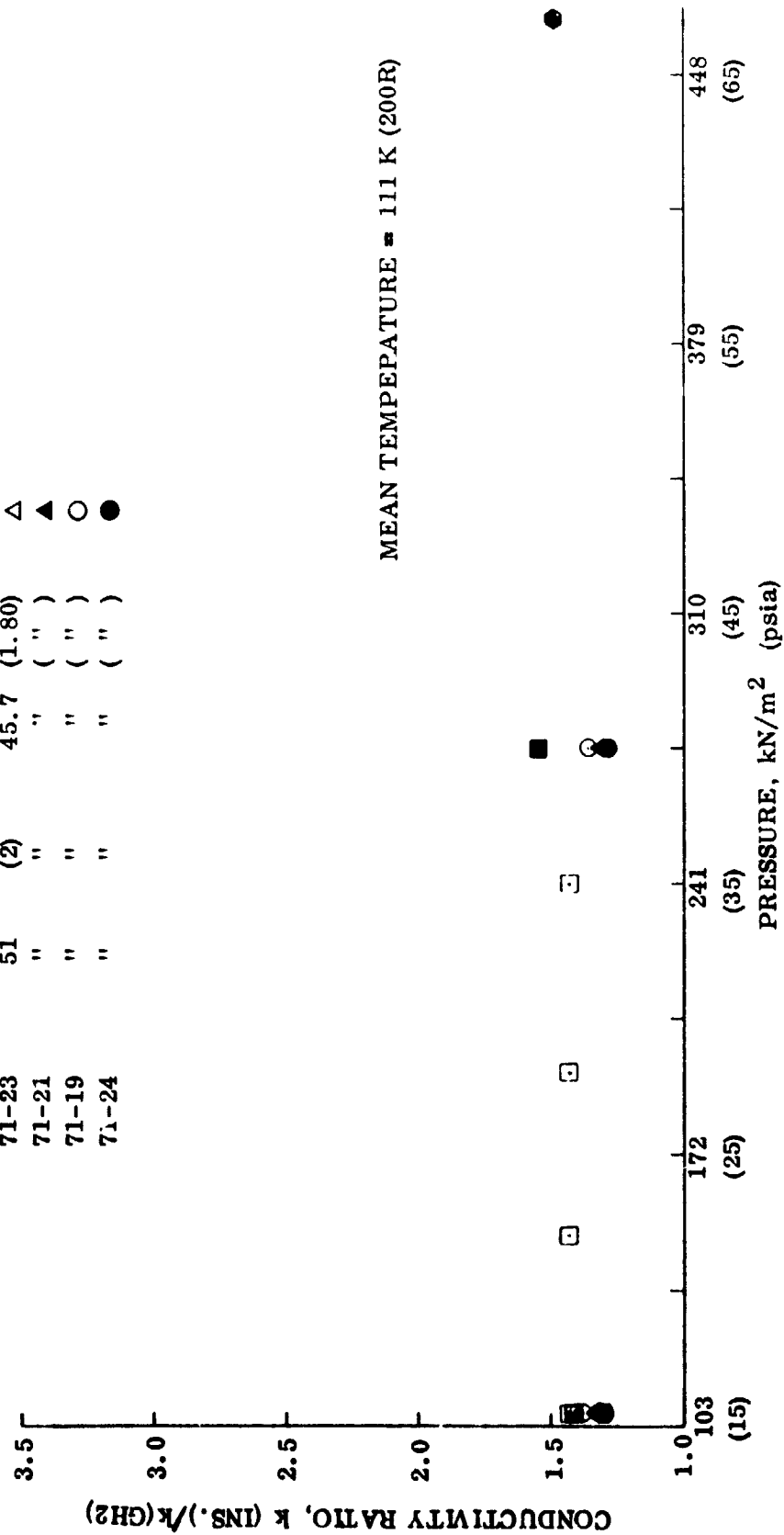


Figure 6-15. PPO Foam Thermal Conductivity in LH_2 , Horizontal Orientation - Cells Vertical

PANEL NO.	PANEL THICK.	TEST THICK.	SYMBOL
-	mm (in.)	mm (in.)	
-	51 (2)	45.7 (1.80)	○
-	51 (2)	45.7 (1.80)	●
71-3	76 (3)	21.6 (0.85)	□
71-4	76 (3)	68.6 (2.70)	■
71-23	51 (2)	45.7 (1.80)	△
71-21	" (")	"	▲
71-19	" (")	"	○
71-24	" (")	"	●

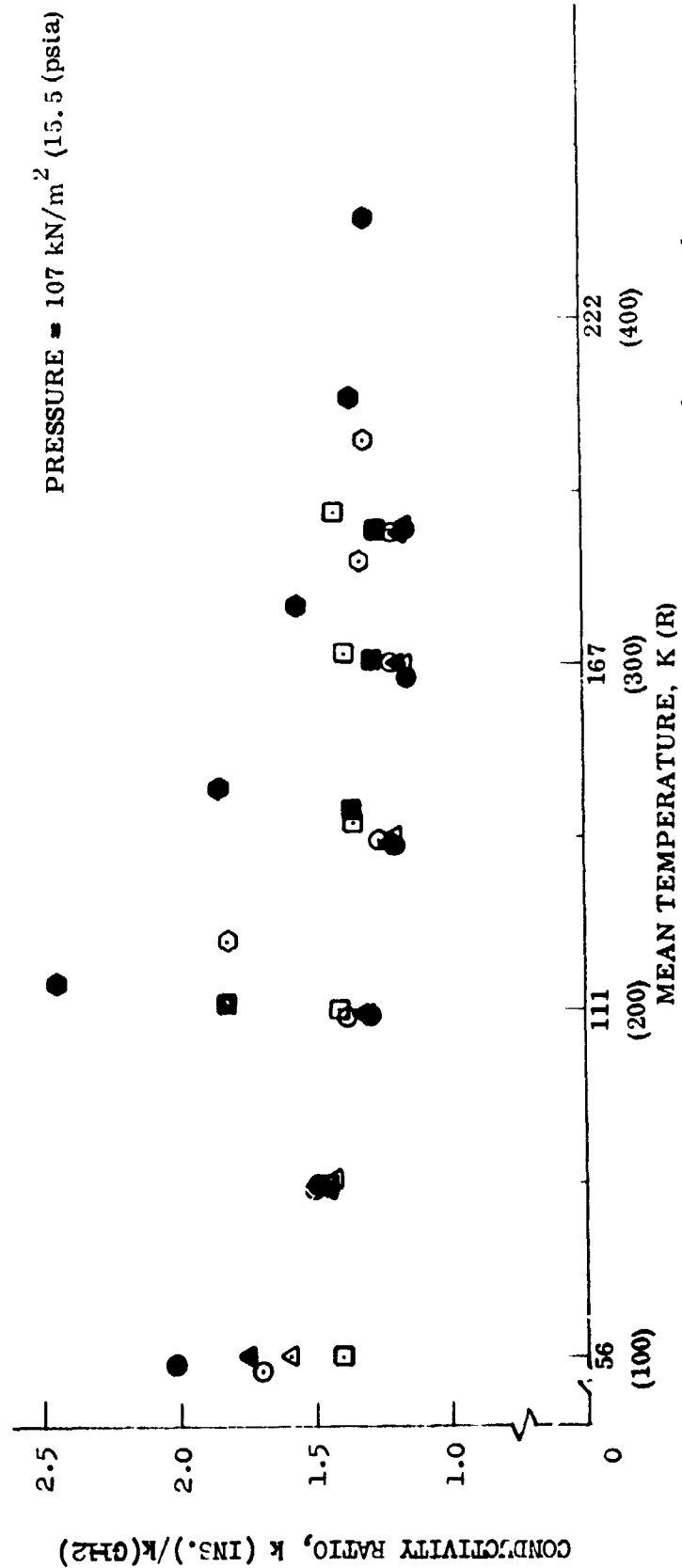
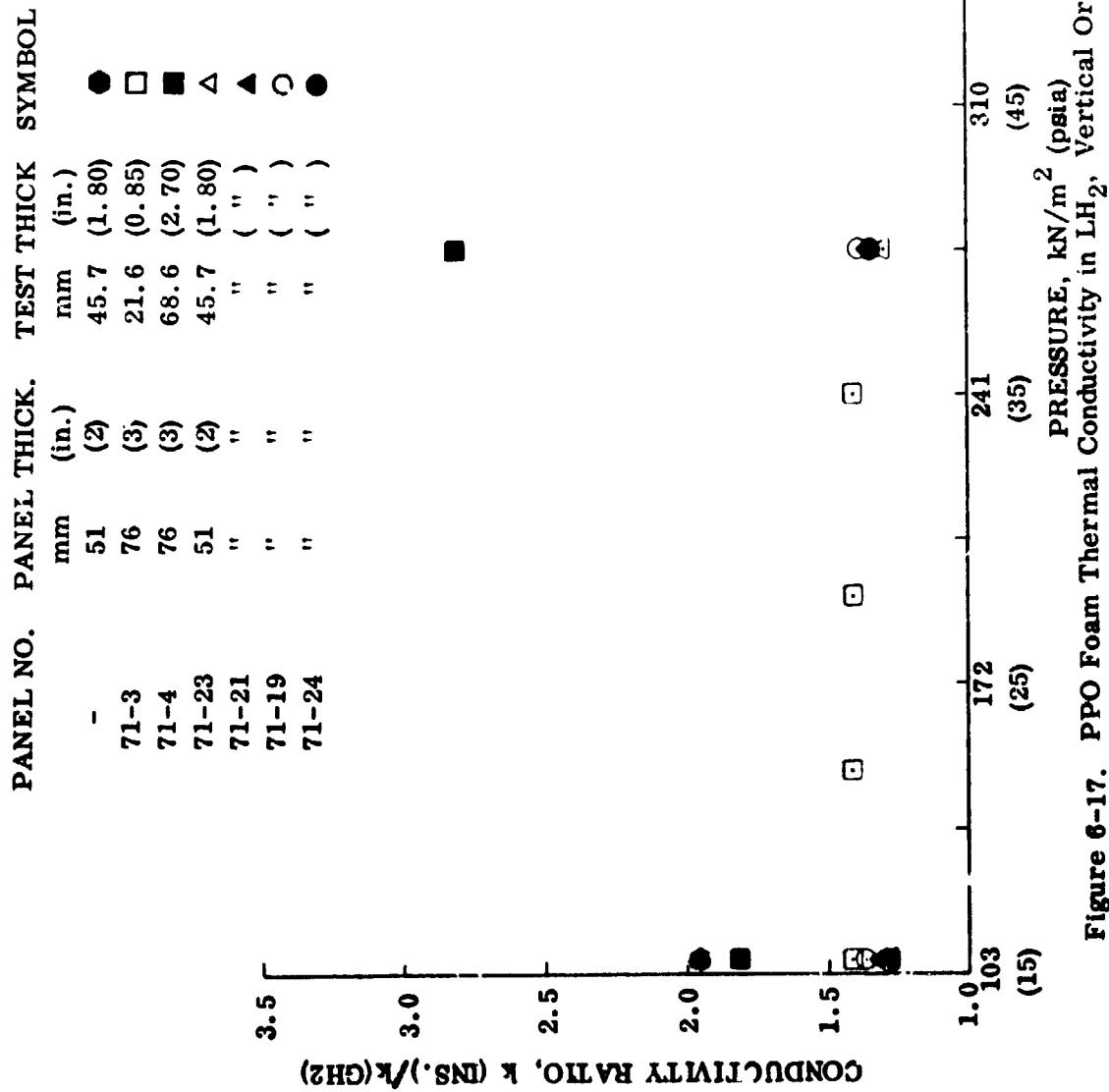


Figure 6-16. PPO Foam Thermal Conductivity in LH₂, Vertical Orientation - Cells Horizontal



MEAN TEMPERATURE = 111 K (200R)

Figure 6-17. PPO Foam Thermal Conductivity in LH_2 , Vertical Orientation - Cells Horizontal

Figure 6-18 and 6-19 show the thermal performance data taken at 107 kN/m^2 (15.5 psia) for the horizontal (cells-vertical) and vertical (cells-horizontal) specimen orientations, respectively. Here the thermal conductivity ratio is plotted as a function of the mean foam temperature. Data for panels 71-19, 21, 23, and 24 are included for comparison. Specimen 71-35 exhibits the worst thermal performance of the eight in both the horizontal and vertical orientations. The second poorest performer in the vertical orientation, specimen 71-30, is the best performer of the eight in the horizontal orientation. In general specimens 71-31 and 33 exhibit conductivity ratios similar to those of specimens 71-19 through 24.

Based on integrating the conductivity ratio curves, a thermal performance ranking was made. These results are listed in Table 6-2. The specimens are ranked based on both the horizontal and vertical orientations, and on a composite of both sets of data. The integrated average thermal conductivity ratios for the panels are calculated between 56 and 183K (100 and 330 R) and ratioed to the lowest value of the eight to indicate the degree of variation between the panels. The composite ratios indicate that the overall performance of the first four panels are essentially identical and that there is not a significant performance difference between the top six panels. Only panels 71-30 and -35 are clearly inferior.

All eight specimens were subjected to pressure transients to evaluate the effect on temperature distributions within the specimen and on thermal performance. The results for specimen 71-33 are shown in Figure 6-20. The gaps in the three temperature curves indicate a period of time when the data acquisition was programmed to continually record only pressure. During the pressure transient the maximum rise rate was 25 kN/m^2 per sec (3.6 psi per sec) and the average value was 17 kN/m^2 per sec (2.4 psi per sec). The pressure transient caused immediate depressions in the temperatures in the foam followed by partial recovery in the two thermocouples

Table 6-2. Thermal Performance Ranking, Formulation Screening Specimens

Specimen	Horizontal		Vertical		Composite	
	Rank*	Ratio	Rank	Ratio	Rank	Ratio
71-21	2	1.030	2	1.001	1	1.000
71-24	4	1.049	1	1.000	2	1.009
71-23	3	1.046	4	1.015	3	1.015
71-19	7	1.092	3	1.014	4	1.036
71-31	5	1.058	5	1.088	5	1.058
71-33	6	1.077	6	1.097	6	1.072
71-30	1	1.000	7	1.546	7	1.264
71-35	8	1.261	8	2.287	8	1.766

*In order of increasing thermal conductivity

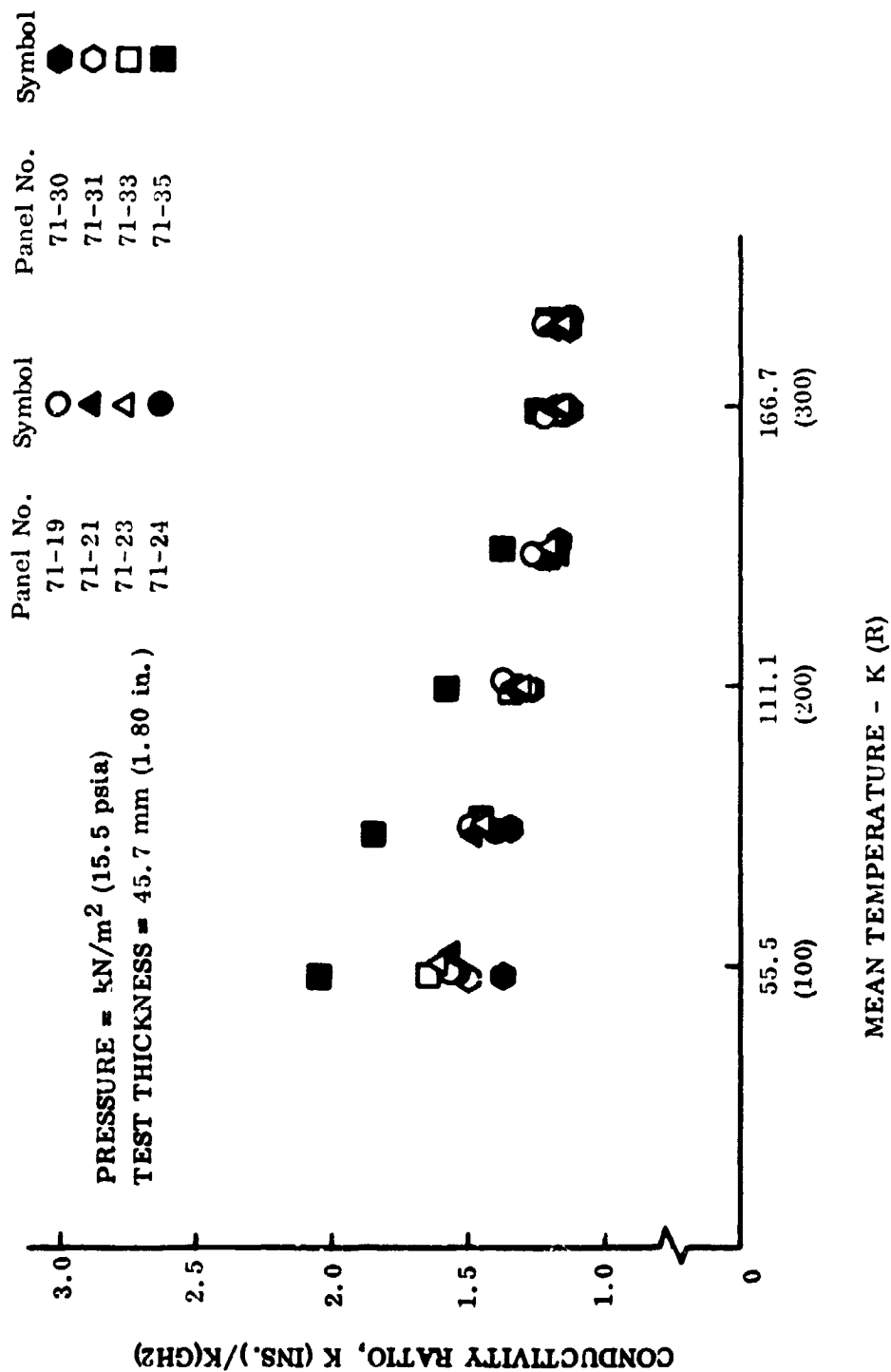
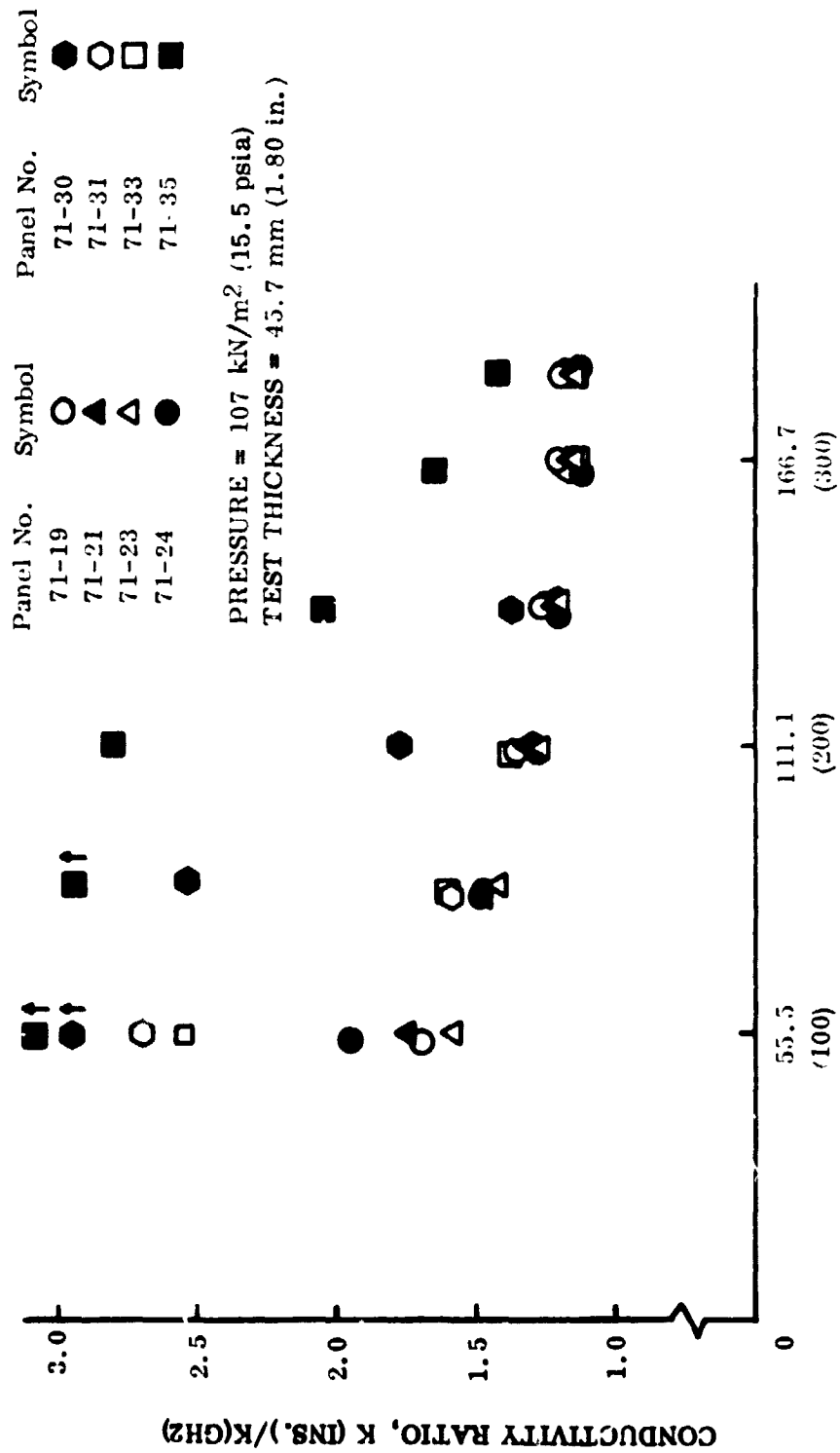


Figure 6-18. Thermal Conductivity in the Horizontal Orientation - Cells Vertical



MEAN TEMPERATURE - DEG. K (DEG. R)

Normal Conductivity in the Vertical
Orientation - Cells Horizontal

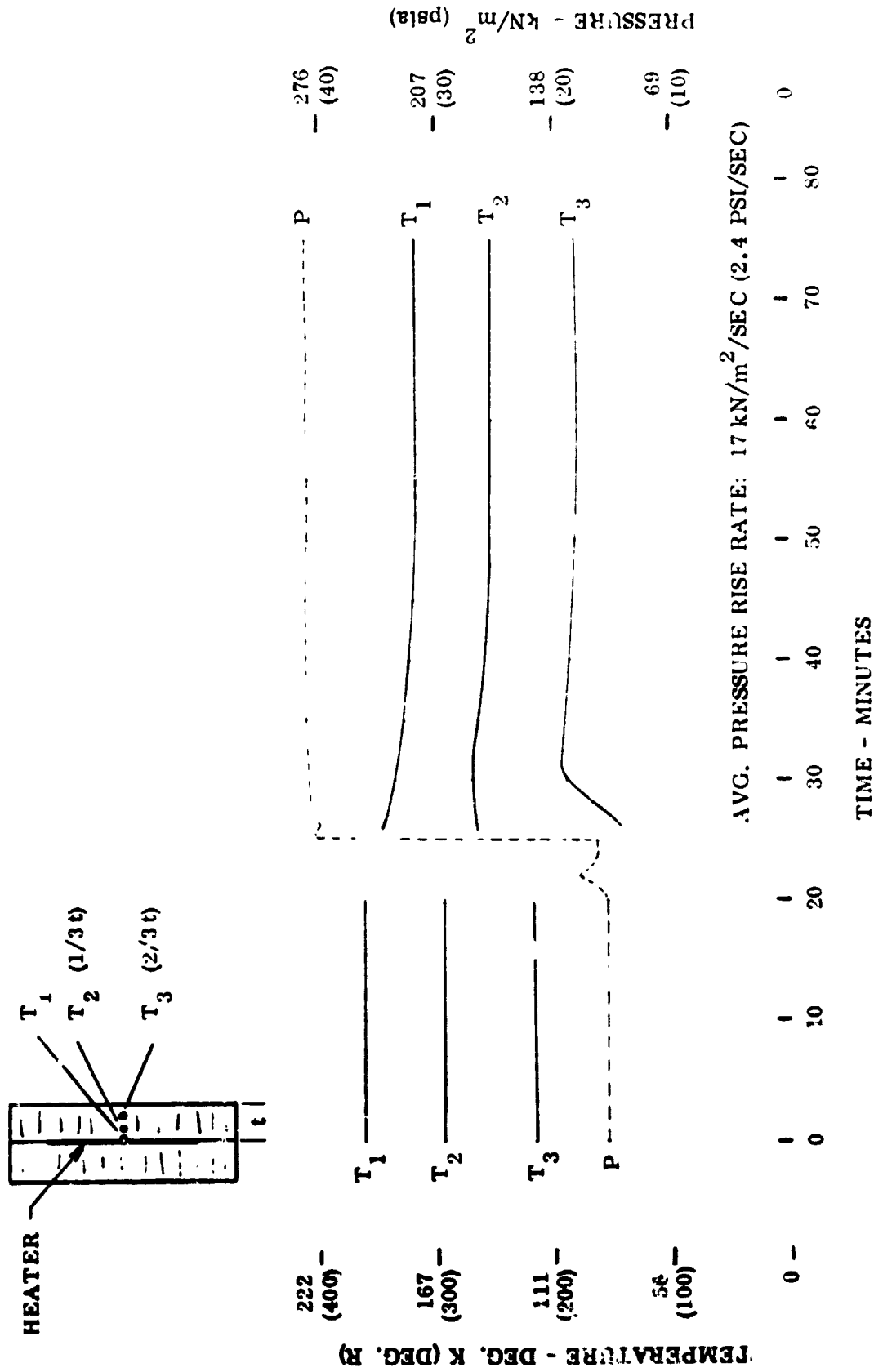


Figure 6-20. Effect of Pressure Rise on Temperature Profile (Specimen 71-33)

nearest the cold face. After fifty minutes at the elevated pressure the temperatures had dropped further and there was no indication that the temperatures would recover completely. Similar data for specimen 71-24 is shown in Figure 6-21. Here the average pressure rise rate was 40.0 kN/m^2 per sec (5.8 psi per sec) and the temperatures recovered to within 11K (20R) of the initial values in approximately 8 minutes. Theoretically, for high quality material, the temperatures should eventually recover completely.

6.2 CONFIGURATION SCREENING

Ten thermal conductivity tests were performed during the PPO foam configuration screening program. The panel numbers, the original (manufactured) and test thicknesses, and the test specimen densities are listed in Figures 6-22 and 23. The dashed lines in these two figures represent the spread of the data for the better formulation screening specimen described above. Thicknesses ranged from 7.1 to 70.0 mm (0.28 to 2.76 in.) and densities from 28.0 to 62.5 kg/m^3 (1.75 to 3.90 lb/ft^3). The first six specimens listed are all nominally 25.4 mm (1.0 in.) thick. The first three were cut from 30 mm (1.18 in.) panels. The purpose of these tests was to determine whether or no significant differences in thermal performance existed between sheets cut from the surface of thicker panels and sheets cut from the center of thinner panels. Comparing specimens of similar density, in every case specimens cut from the surface of a 75 mm (3 in.) panel exhibited superior thermal performance. This is consistent with the observation that the cell structure near the surface of a thick panel is more uniform and of higher quality than that in the center of a thin panel.

Two panels thicker than 24.5 mm (1.0 in) were tested, a 46.0 mm (1.8 in) and a 70.0 mm (2.76 in) panel. The 46.0 mm (1.8 in) panel exhibited a performance similar to that of the better 25.4 mm (1.0 in) panels. The 70.0 mm (2.76 in) panel also performed as well except at the lowest hot wall temperature, 89K (160R) (mean temperature 56K (100R)). At this lowest temperatures, the LH_2 - GH_2 interface moved into the foam cells, as indicated by thermocouples in the foam, reducing the gas layer thickness. Since the effective conductivity of the PPO foam is based upon the total foam thickness, the effective conductivity increases. To efficiently utilize the 70 mm panel, the hot wall temperature would have to be increased to about 200K (360R) to maintain the LH_2 - GH_2 interface at the foam surface.

Comparing the performance of the 14.5 mm (0.57 in) and 7.1 mm (0.28 in) thick panels with the 25.4 mm (1.0 in) panels, as the panel thickness is reduced, the thermal performance worsens. The 14.5 mm (0.57 in) specimen has a very poor performance and the 7.1 mm (0.28 in) specimen performed so poorly that it was not possible for the heater to bring the hot face temperature above 278K (500R). The minimum effective thickness for this open-cell insulation is between 25.4 mm (1.0 in) and 14.5 mm (0.57 in).

7.2

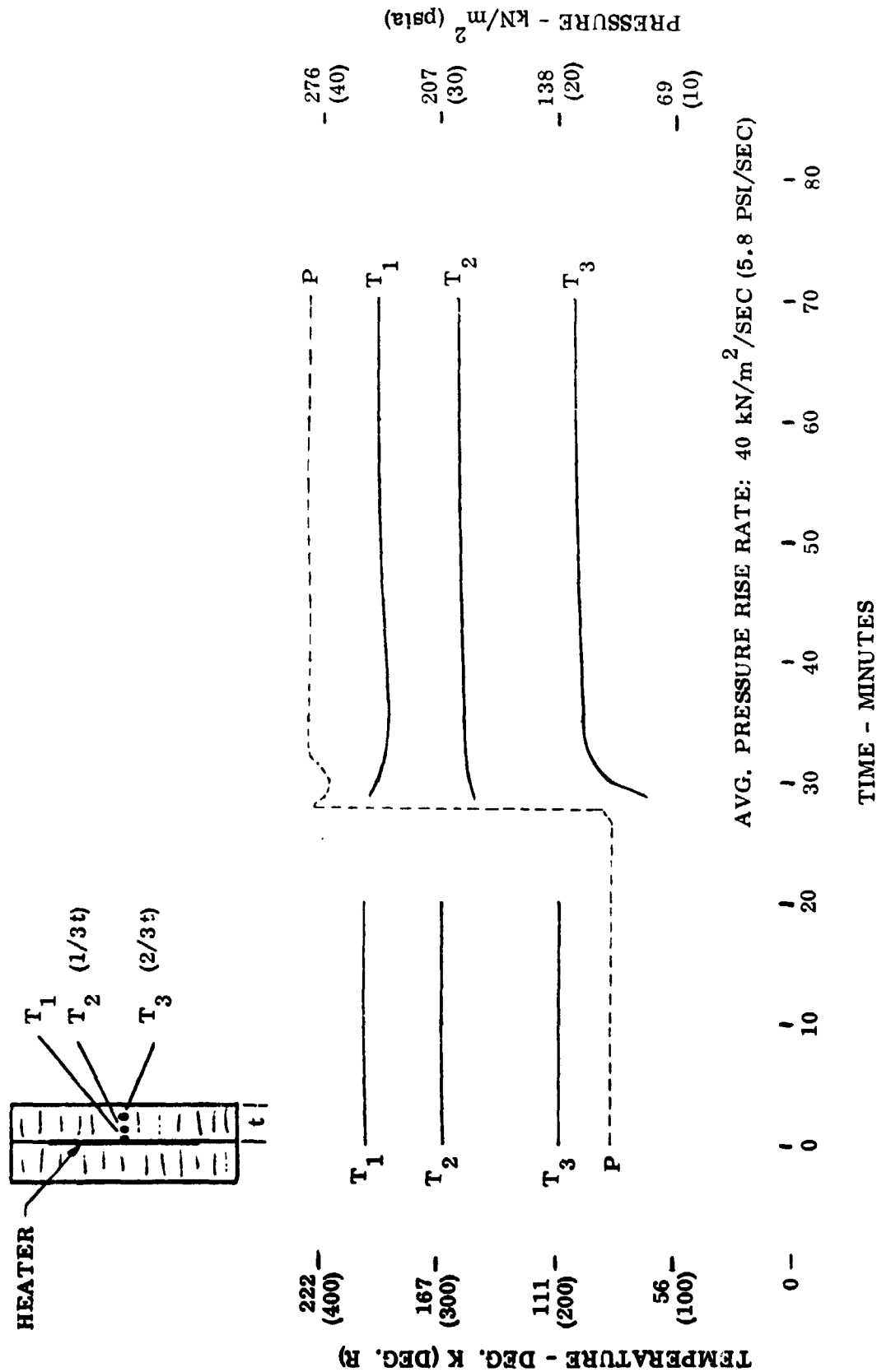


Figure 6-21. Effect of Pressure Rise on Temperature Profile (Specimen 71-24)

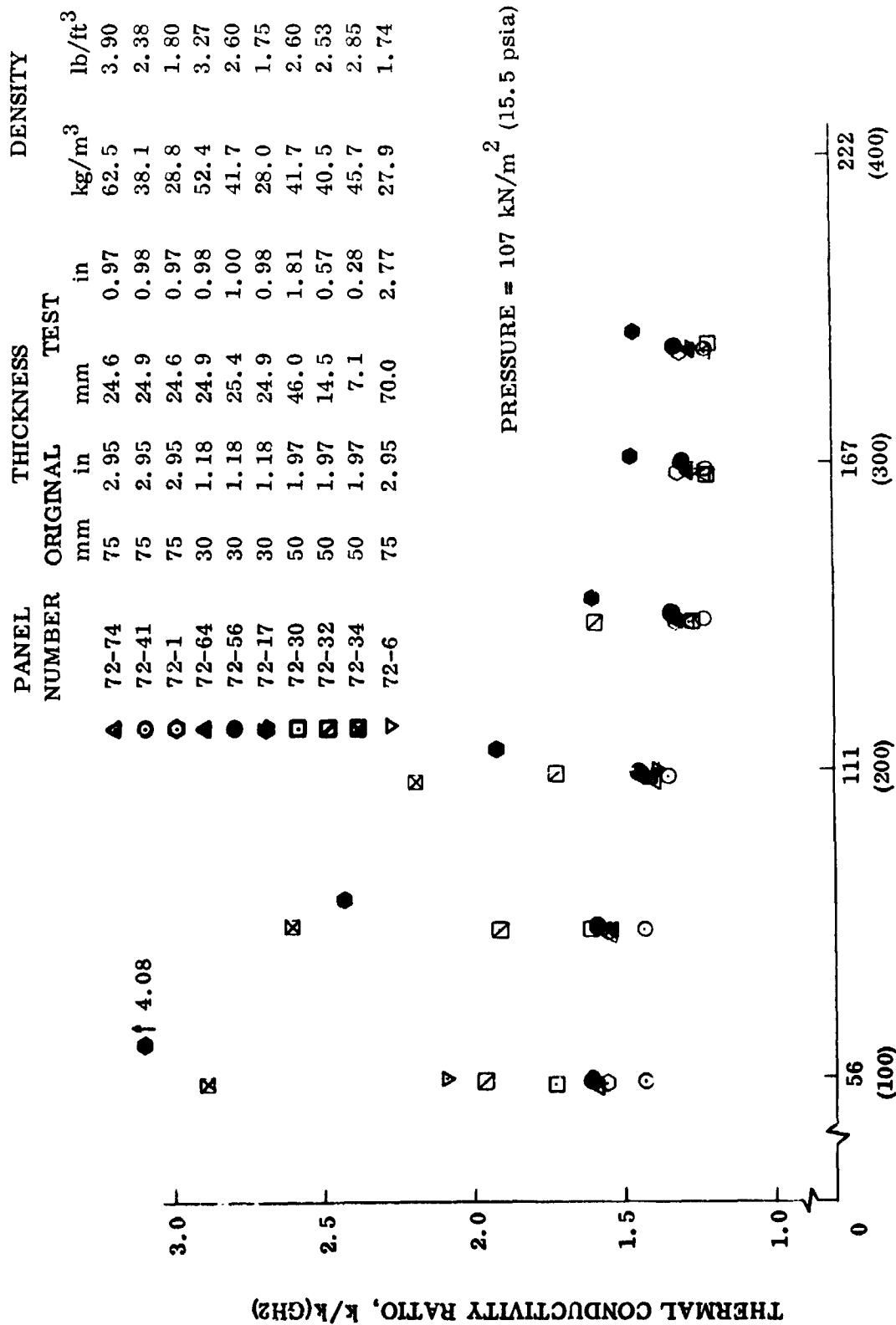


Figure 6-22. Thermal Conductivity in Liquid Hydrogen,
Horizontal Orientation - Cells Vertical

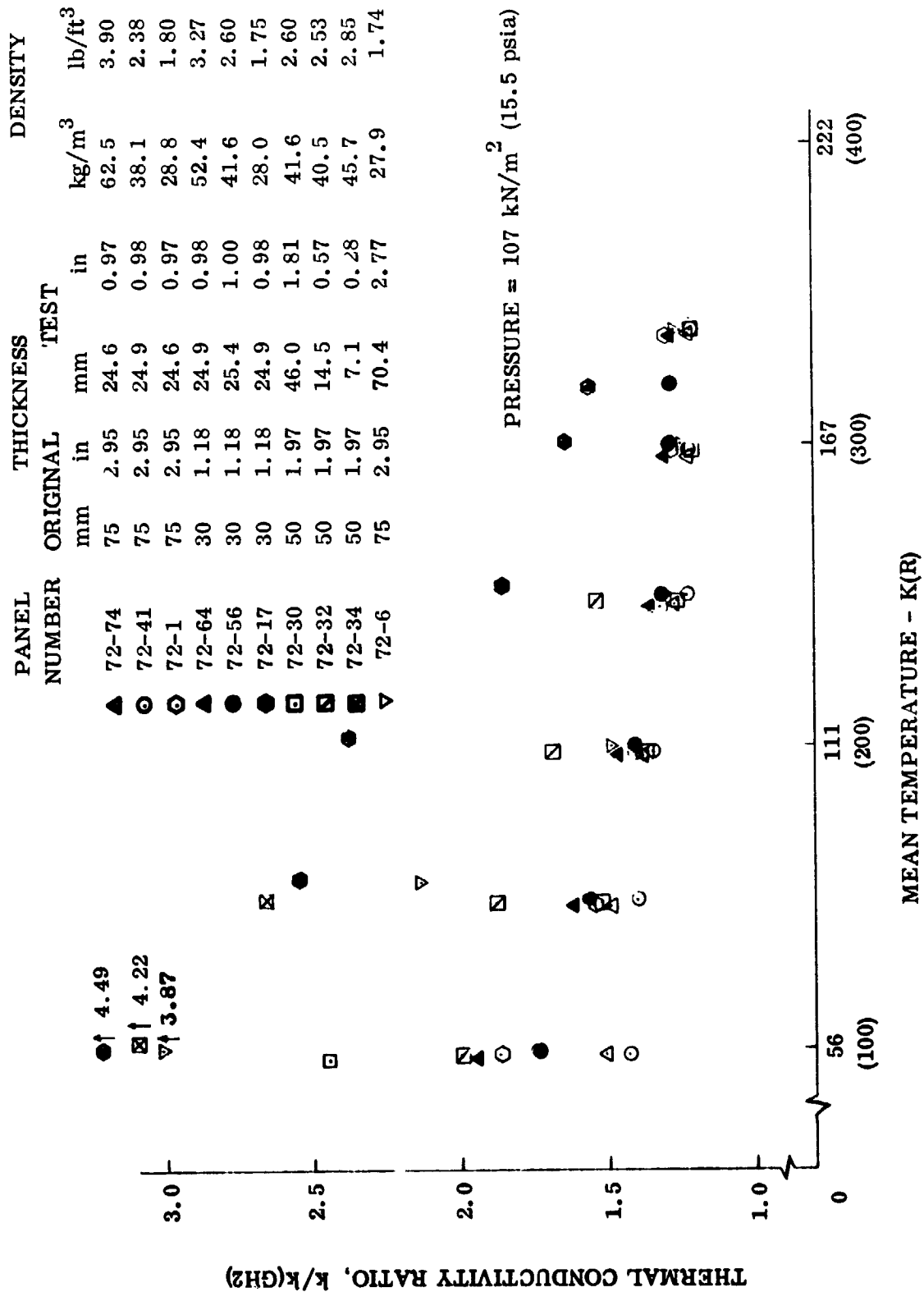


Figure 6-23. Thermal Conductivity in Liquid Hydrogen,
Vertical Orientation - Cell Horizontal

The six 25.4 mm (1.0 in) panels have been ranked based on average thermal conductivity ratios calculated by integration between the mean specimen temperatures of 56 and 188K (100 and 338R). The results are shown in Table 6-3. The three specimens cut

Table 6-3. Thermal Performance Ranking,
Configuration Screening Specimens

Specimen	Horizontal		Vertical		Composite	
	Rank*	Ratio	Rank	Ratio	Rank	Ratio
72-41L	1	1.000	1	1.000	1	1.000
72-74L	2	1.047	2	1.028	2	1.038
72-1L	4	1.078	3	1.094	3	1.086
72-56	5	1.095	4	1.098	4	1.097
72-64	3	1.075	5	1.134	5	1.104
72-17	6	1.566	6	1.854	6	1.710

*In order of increasing thermal conductivity

from the edges of 75 mm (2.95 in.) panels rank highest. This again supports the contention that higher quality material is found at the surface of thicker panels. Another fact to be noted from the data is that within each of the two sets (41L, 74L, 1L and 56, 64, 17) the middle density specimen ranks at the top followed in order by the higher and lower density specimens.

The effect of a rapid increase in tank pressure on the thermal performance of the foam has been investigated. Figure 6-24 illustrates the effect on the temperature profile through the foam specimen. The pressure spike causes an immediate depression in the temperatures, the effect being more pronounced as the distance from the hot face increases. The fast pressure rise causes the liquid/vapor interface to move into the foam equalizing the pressures and cooling the surrounding material. Liquid then vaporizes increasing the mass of gas in the cell and the interface to move back to its more stable position at the surface of the foam. The temperatures then recover almost to the initial value. Table 6-4 shows the average pressure rise rates imposed on the eight specimens, the resulting increase in the conductivity ratio twenty minutes after completion of the pressure rise, and the degree of recovery of the hot face temperature (T_1) after twenty minutes time. Once again the three specimens representing the edge material of thick foam panels exhibit superior performance. The 70.0 mm (2.76 in) panel, specimen 72-6, showed evidence of severe LH_2 intrusion and little temperature recovery even though the pressure rise was quite slow. This may be due to the low quality of the 70.0 mm (2.76 in) foam which

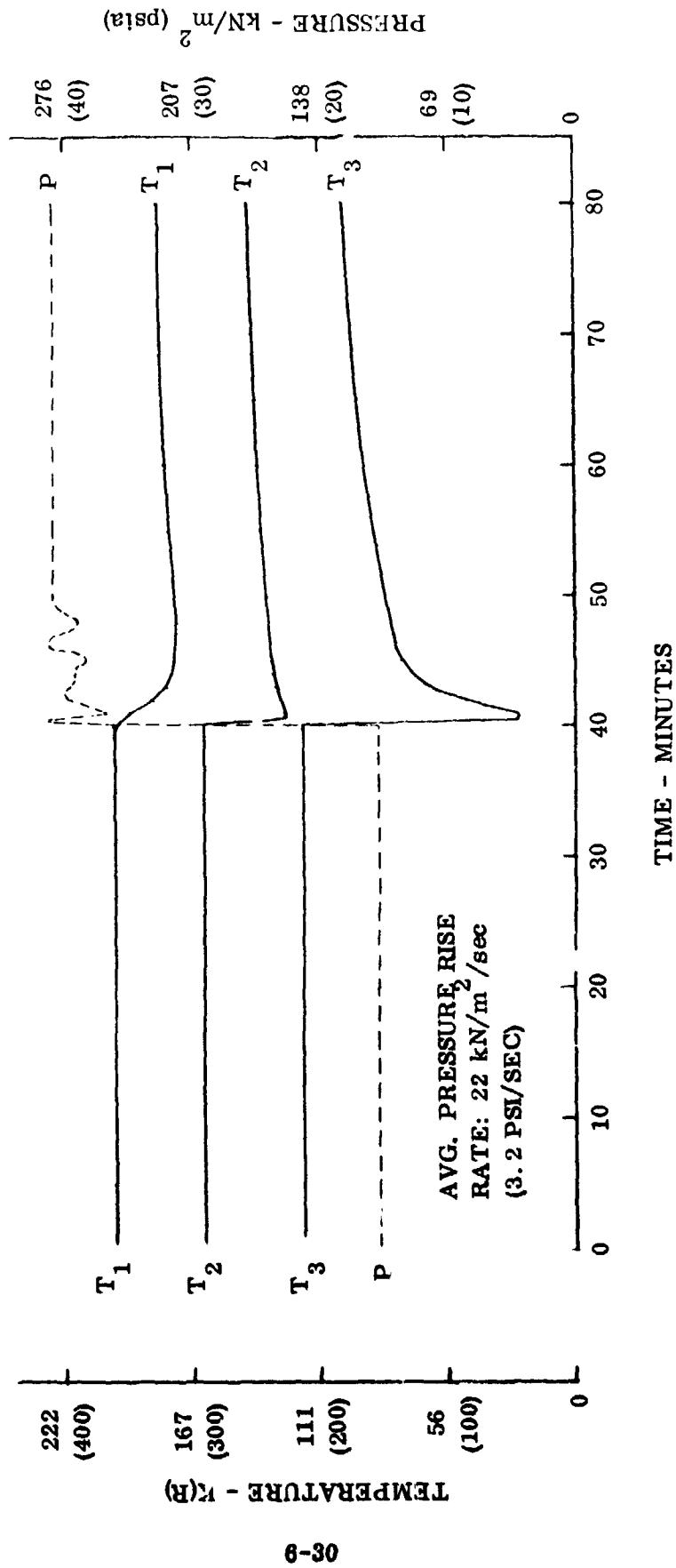


Figure 6-24. Effect of Pressure Rise on Temperature Profile (72-30).

had a porous low density center layer. When the liquid-gas interface reaches the center region, the interface becomes unstable, allowing excessive liquid intrusion.

Table 6-4. Effect of Pressure Rise

Specimen	Avg. Pressure Rise Rate ⁽¹⁾ kN/m ² /sec (psi/sec)		Conductivity Increase ⁽²⁾ %	Hot Face Temperature Recovery ⁽²⁾ %
72-74L	17	(2.4)	0	100
72-41L	20	(2.9)	0	100
72-1L	17	(2.5)	0	100
72-64	21	(3.0)	7	94
72-56	25	(3.6)	8	93
72-17	26	(3.7)	64	66
72-30	22	(3.2)	15	89
72-32L	19	(2.7)	2	99
72-34L	8.3	(1.2)	15	71
72-6	1.2	(0.17)	62	67

(1) Between 107 and 276 kN/m² (15.5 and 40 psia).

(2) Twenty minutes after completion of pressure rise.

6.3 FINAL PRODUCTION TEST

Nine Class II panels 50 mm (1.98 in) thick and one acceptance test panel, where the density gradients were measured, were received from the vendor in April 1975. The panels were x-rayed and paper removed to Class I configuration. They were also inspected visually and on a light table. The best of these panels, 75-10, was selected for a thermal conductivity test. The final test data is shown in Figure 6-25. Both the vertical and horizontal orientation were tested. The lines plotted on the graph, Figure 6-25, represent the lowest thermal conductivity data from all previous tests. These final panels represent a significant improvement in the thermal conductivity of PPO foam in LH₂. This is attributed primarily to the improved uniformity of the foam wherein the density gradient parallel to the fibers has been kept within the specified value, $\pm 10\%$. This tends to minimize cell size in the middle of the foam thus reduce the effects of gas convection.

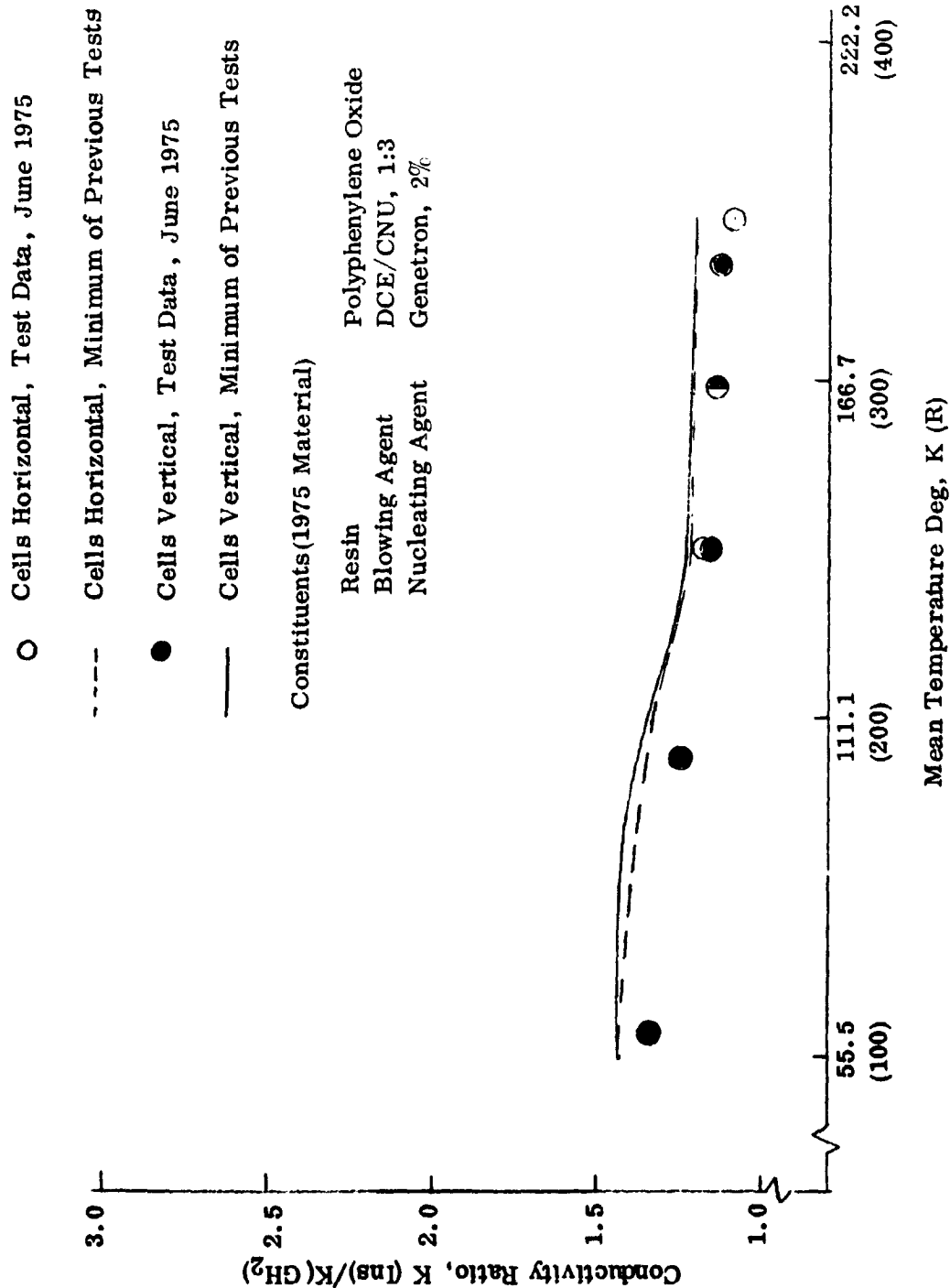


Figure 6-25. Final Production Thermal Conductivity Test Results

SECTION 7

GEOMETRIC PROPERTIES

7.1 PERMEABILITY

One of the parameters which is useful in the evaluation of the internal structural characteristics of PPO foam is the material permeability. Permeability measurements can be used together with mechanical strength and thermal conductivity data to screen different foam configurations and predict structural and thermal performance in an actual tank usage situation. Theoretically a good open cell insulation material should have a high permeability in the direction parallel to the cell orientation and a low permeability laterally. A low lateral permeability curtails convection currents in the tank sidewall insulation while a high parallel permeability reduces the chance of insulation failure due to pressure changes in the cells. Permeability test data was correlated with thermal performance of specimens from the same foam panel.

7.1.1 TEST APPARATUS. The techniques used to evaluate permeability is the measurement of the internal resistance of the material to gas flow, determined by recording pressure drop through the material as a function of gas flow rate, both parallel (longitudinal) and perpendicular (lateral) to the cell orientation. Detailed analysis of this data for different foam configurations can lead to conclusions about the relative bulk densities, the comparative number of blocked cells, the extent of cell interconnections, and the relative mean cell diameters. If this information can be correlated with thermal and mechanical strength data, the material configuration can be adequately characterized with respect to its adaptability to specific use situations, and the effects of variations in the internal structure on material strength and thermal performance can be predicted.

The apparatus for testing PPO foam specimens in the perpendicular direction is illustrated in Figure 7-1. This apparatus consists of three aluminum plates with very soft rubber bonded to one side. The 88.9 mm (3.50 in.) by 44.5 mm (1.75 in.) foam specimen is bonded, with silicone rubber, between Plexiglas plates, Figure 7-2, and is then clamped between the top and bottom plates of the test apparatus. Then the end plate, with fittings for the manometer and inlet, is mounted.

Two apparatus were used to test PPO foam specimens in the parallel direction. The first, illustrated in Figure 7-3, utilized 14.1 cm (5.55 in.) diameter foam discs as specimens. The cylindrical test beaker was fabricated from 6.35 mm (0.25 in.) thick Plexiglas plate. Very soft (15-30 Durometer) silicone rubber seals are used to prevent leakage around the test specimen. The test specimen is held in place with a

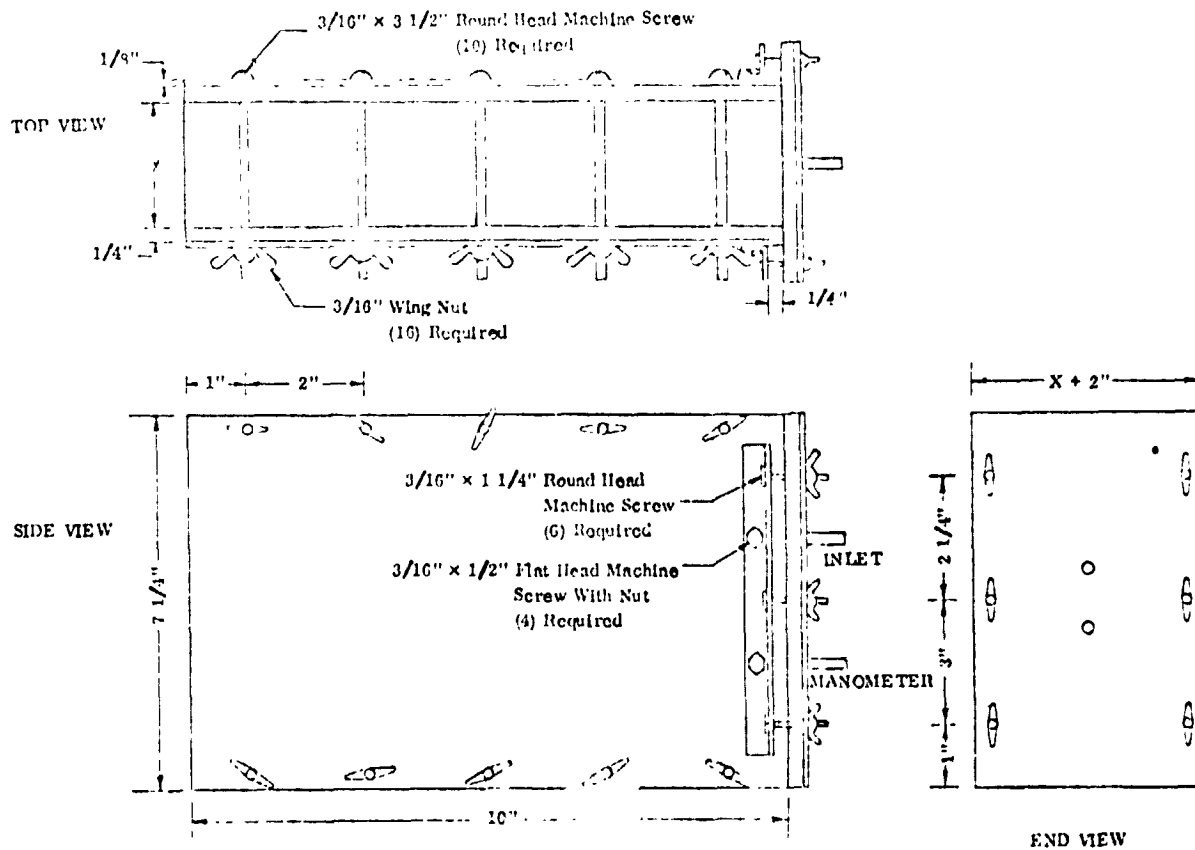


Figure 7-1. Perpendicular Gas Flow Resistance Apparatus



Figure 7-2. Specimens Prepared for Perpendicular Gas Flow Resistance Test

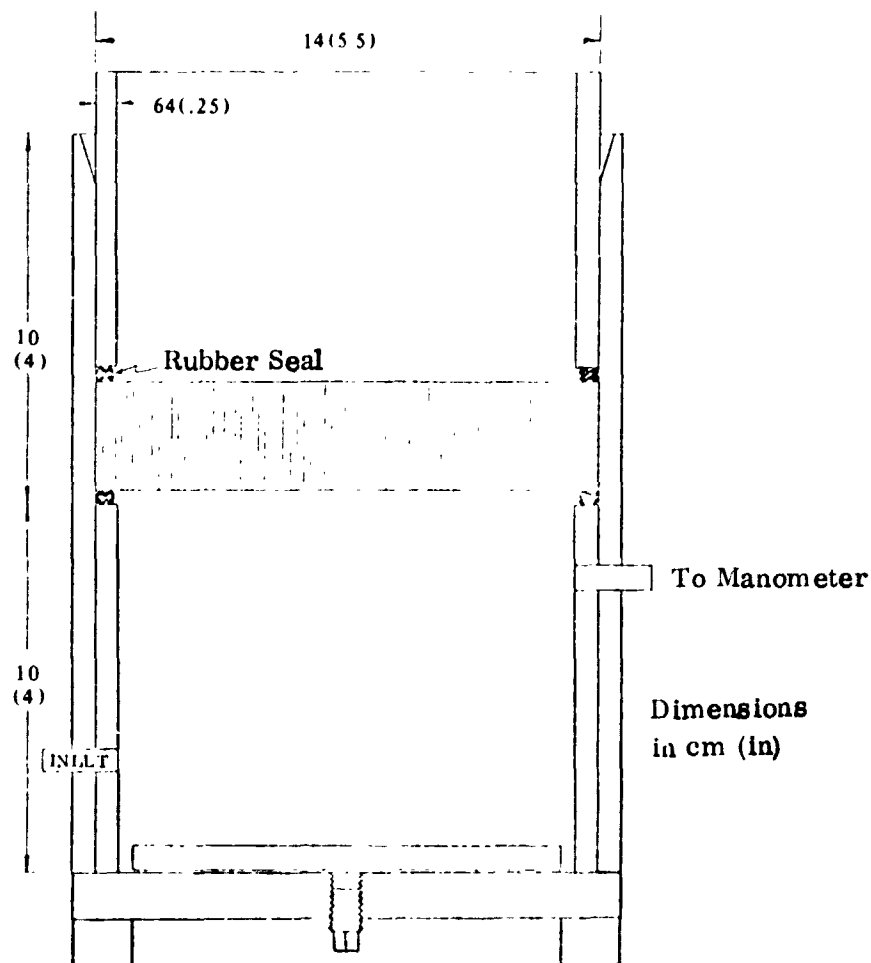


Figure 7-3. Cylindrical Beaker Parallel Gas Flow Resistance Apparatus

section of tubing weighted down with about 9 kg (20 lb) of lead. A taper at the top of the beaker is required to allow the specimen to be placed in the beaker with ease since PPO foam specimens are about 1.27 mm (0.050 in.) oversize. To remove a specimen, the plug at the bottom of the beaker is removed and the plate in the bottom of the beaker is forced against the specimen with a rod.

The second parallel flow apparatus, illustrated in Figure 7-4, was designed to allow both parallel and perpendicular flow tests on the same piece of foam. After testing in the perpendicular flow apparatus, Figure 7-2, the Plexiglas side plates are trimmed even with the foam block and then the remaining pieces of the side plates are bonded, with silicone rubber, to the 88.9 mm (3.50 in.) faces of the foam block, see Figure 7-5. The test specimen is then placed on top of the test apparatus and is compressed, by lead weights, against a soft rubber seal bonded to the top rim of the

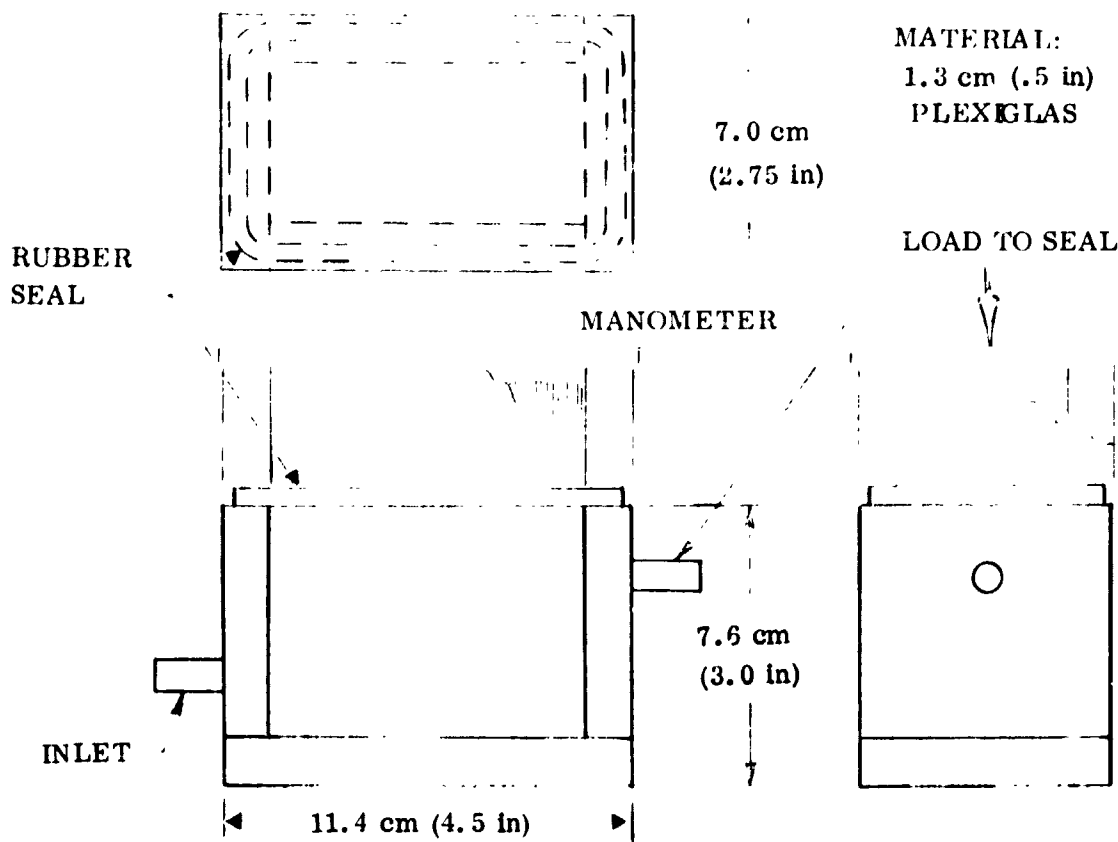


Figure 7-4. Square Beaker Parallel Gas Flow Resistance Apparatus

rectangular beaker. Thus, a positive seal is established between the beaker and the Plexiglas frame around the foam, avoiding any crushing of the foam specimen.

A schematic of the gas flow resistance test setup is shown in Figure 7-6. The bleed valve allows the gas supply regulator valve to operate stably at a high flow rate, while the flow to the test apparatus is quite low. The multiple tube flowmeter and the water and mercury manometers allow measurements to be taken over a very wide range of flow rates and pressures. Manometer readings were sighted with a cathetometer. A photograph of the test setup is shown in Figure 7-7.

Parallel gas flow tests have been performed on eight of the 71- panels using the cylindrical test beaker and two specimens for each panel. The test results are shown in Figure 7-8. The large variance in the gas flow resistance of the specimens is indicative of large differences in the internal structure of the various panels. An indication of the dispersion within a panel is provided by Figure 7-9 where the test results for both specimens of panels 71-23, -30 and -33 are plotted.

A new parallel flow apparatus was devised for testing specimens from the 72- panel master order using the square beaker apparatus, Figure 7-1. Both parallel and perpendicular flow tests can be performed on the same foam specimen giving more

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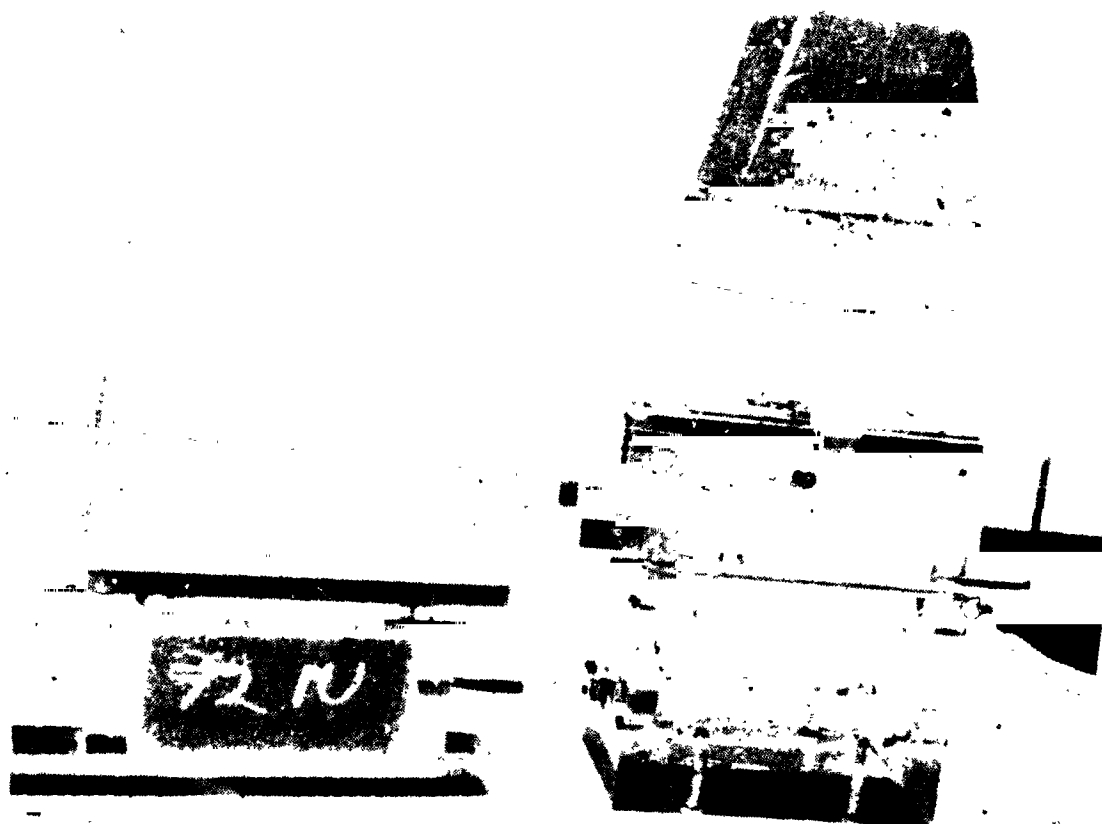


Figure 7-5. Specimen Prepared for Parallel Gas Flow Resistance

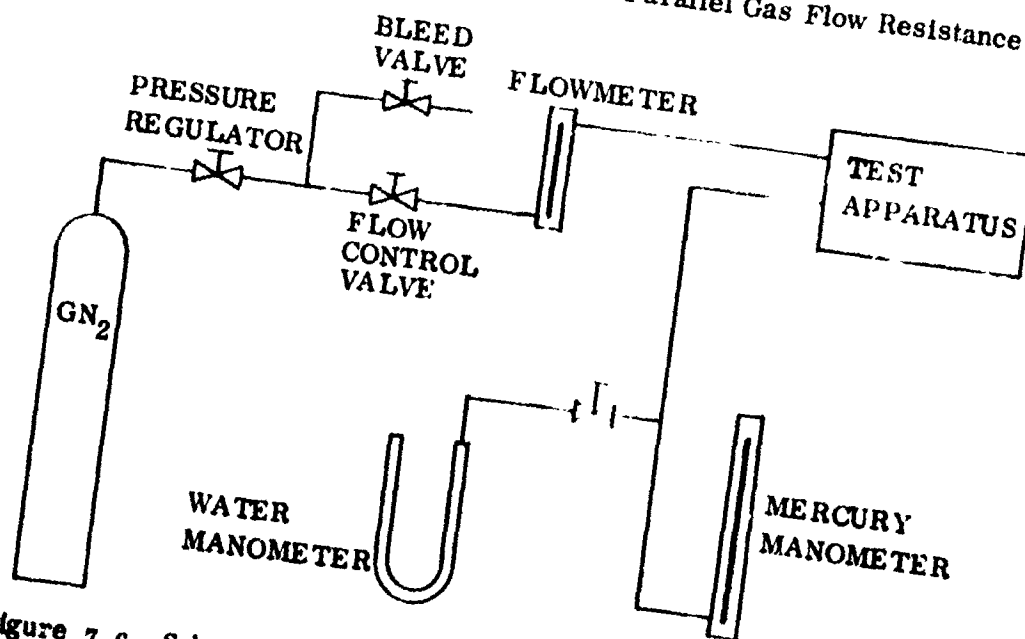


Figure 7-6. Schematic of Gas Flow Resistance Test Setup



Figure 7-7. Gas Flow Resistance Test Setup

consistence data. Also, two test specimens from each panel were taken from adjacent locations in order to minimize variation between specimens. The results of the gas flow resistance tests on the 72- panels are presented in Table 7-1.

All of the panels in Table 7-1 exhibit a low flow resistance in the direction parallel to the foam cells. In the perpendicular direction, the panels with a uniform appearance, panels 72-1, 34, 41 and 74, have a high flow resistance. Panel 72-60 has a severely curved cell structure and panels 72-18 and 55 have a low density center layer. The 2.54 cm (1.0 in.) panels cut from the surface of a 7.62 cm (3.0 in.) panel, such as panels 72-1, 41 and 74, have a more uniform appearance and better flow resistance than panels formed to their full thickness, such as panels 72-18, 55 and 60. Panel 72-34 was cut from the edge of a 7.62 cm (3.0 in.) panel, but it is only 0.76 cm (0.30 in.) thick. A comparison of the gas flow resistance data with the bulk density variations reveals no discernible correlation. Although no relation between

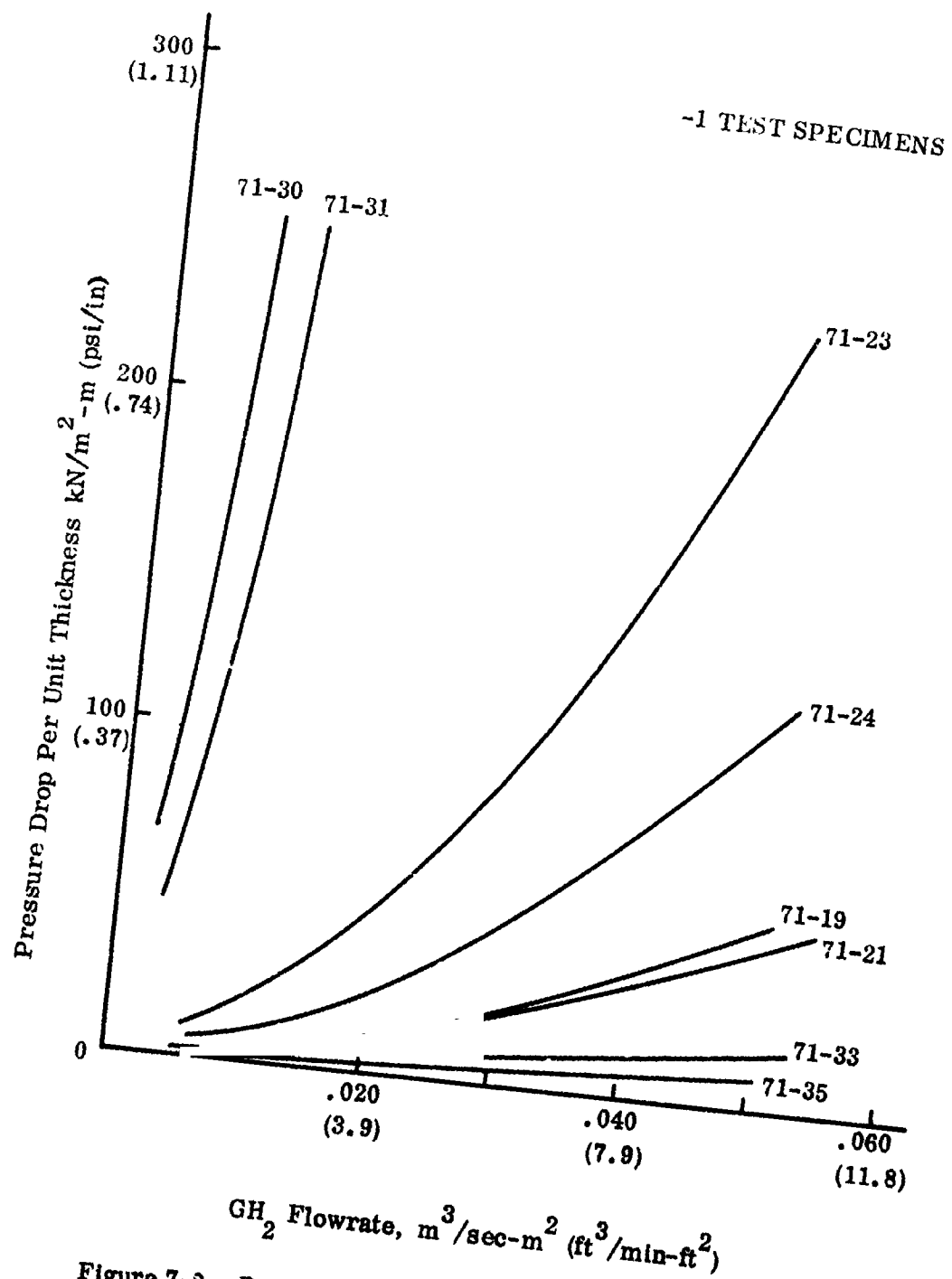


Figure 7-8. Parallel Permeability Test Results

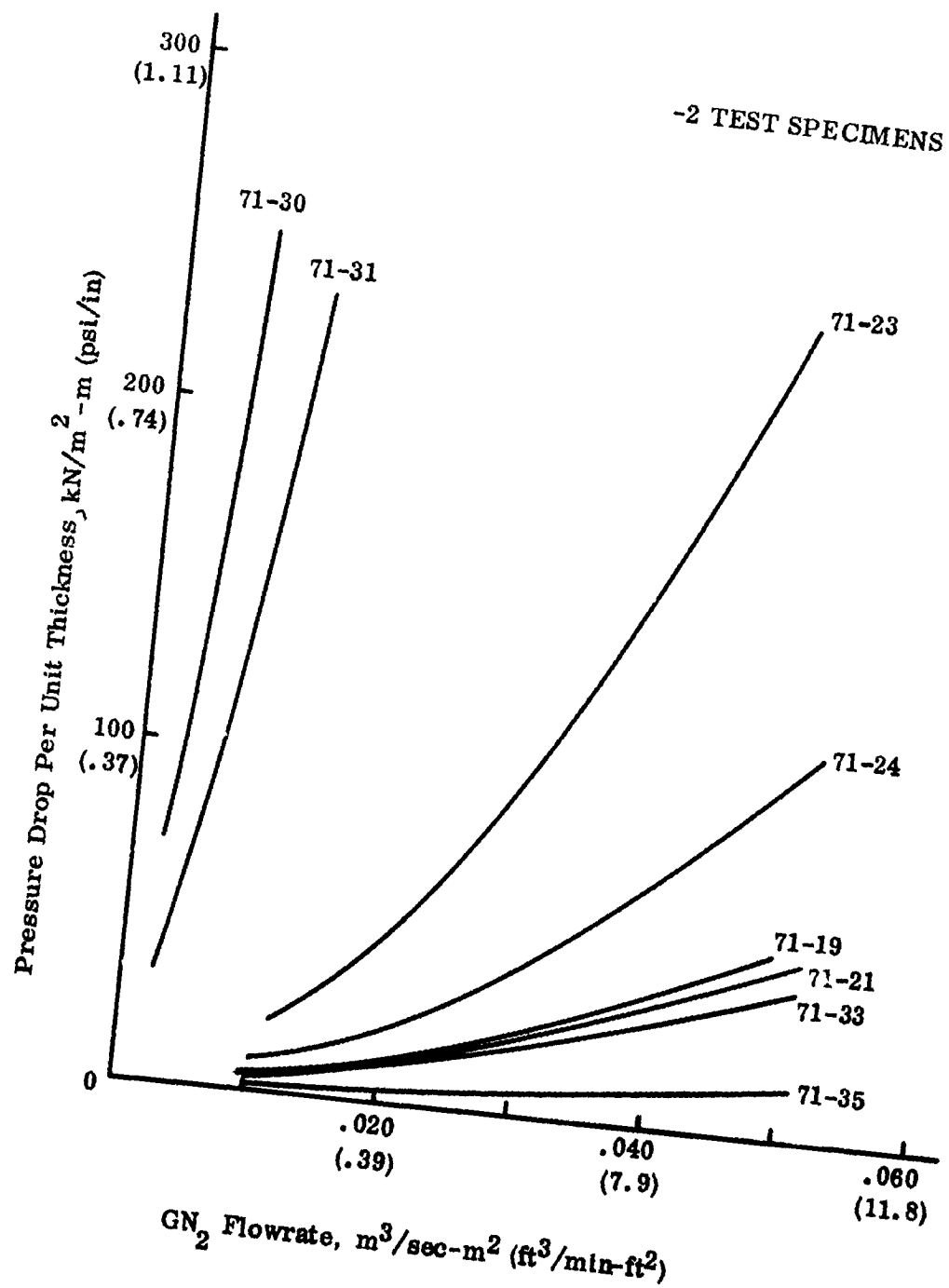


Figure 7-8. Concluded

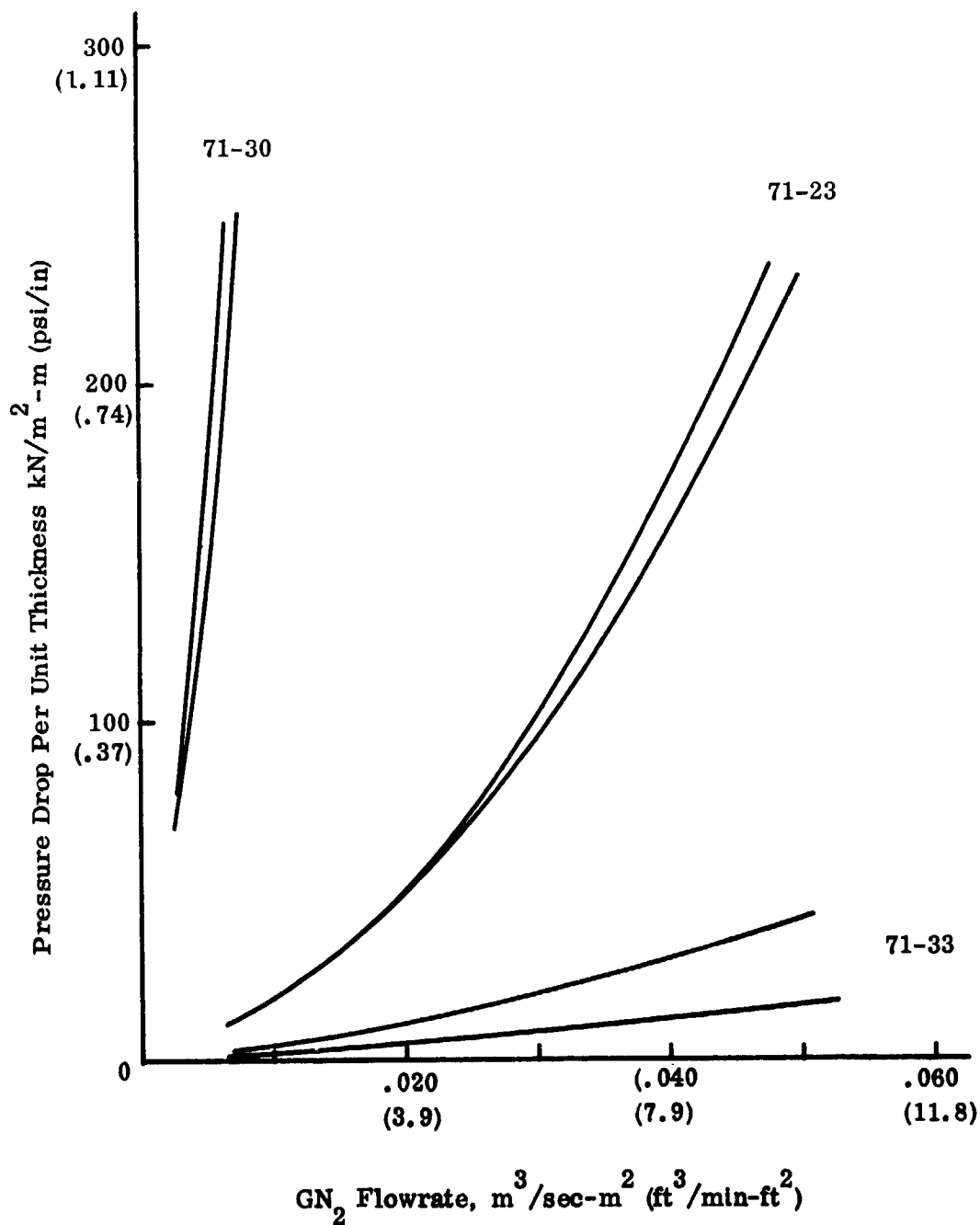


Figure 7-9. PPO Foam Permeability for samples Cut from the Same Thermal Conductivity Specimens

Table 7-1. Gas Flow Resistance

Panel	GN ₂ Gas Flow Resistance kN sec/m ⁴ (psi/in)/(ft ³ /min-ft ²)			Ratio	Thickness cm (in.)	
	Perpendicular		Parallel			
72-41	3911 (0.0732)		1026 (0.0192)	3.8	2.54	(1.0)
72-74	6304 (0.118)		581 (0.0109)	10.8	2.54	(1.0)
72-1	4381 (0.0820)		1159 (0.0217)	3.8	2.54	(1.0)
72-18	1143 (0.0214)		310 (0.00580)	36.9	2.54	(1.0)
72-55	930 (0.0174)		104 (0.00195)	8.9	2.54	(1.0)
72-60	3489 (0.0653)		641 (0.0120)	5.4	2.54	(1.0)
72-32	818 (0.0153)		153 (0.00286)	5.4	1.50	(0.59)
72-29	5744 (0.108)		784 (0.0147)	7.3	4.60	(1.81)
72-3	4291 (0.0803)		1490 (0.0279)	2.9	7.16	(2.82)
72-34	449900 (8.42)		212 (0.00397)	2120	0.76	(0.30)

thermal conductivity and parallel flow resistance could be found, panels with a high perpendicular flow resistance generally have a better thermal performance (see Section 7.2).

Figure 7-10 illustrates the variation of gas flow resistance within a panel. Panels were sliced into sheets and then tested for permeability perpendicular to the foam cells. From Figure 7-10, it is seen that the middle layer in a panel accounts for the low perpendicular gas flow resistance of the foam. This correlated exactly with the density variations in a panel (see Section 4.2) where the middle layer of a panel is less dense than the outer surface layers.

7.2 FLUID-THERMAL CORRELATION

Results of the thermal conductivity tests have been compared with the results of the density gradient investigation, X-ray inspection and pressure drop tests to determine if any of these techniques could be employed to screen incoming panels before using them in an insulation system. Pressure drop tests and density gradient measurements are destructive test techniques while X-ray inspection is non-destructive.

Ten panels of the 72- panel master order were tested for thermal conductivity. Six of the ten were 25.4 mm (1.00 in) thick and can be compared among themselves. The other four panels tested ranged in thickness from 7.6 mm (0.30 in) to 71.6 mm (2.82 in). The first three panels listed in Table 7-2 were cut from the surface of 75 mm (2.95 in) panels while the last three were originally formed as 25.4 mm (1.0 in) panels. The thermal performance ranking of the six 25.4 mm (1.00 in) panels is given in Table 7-2.

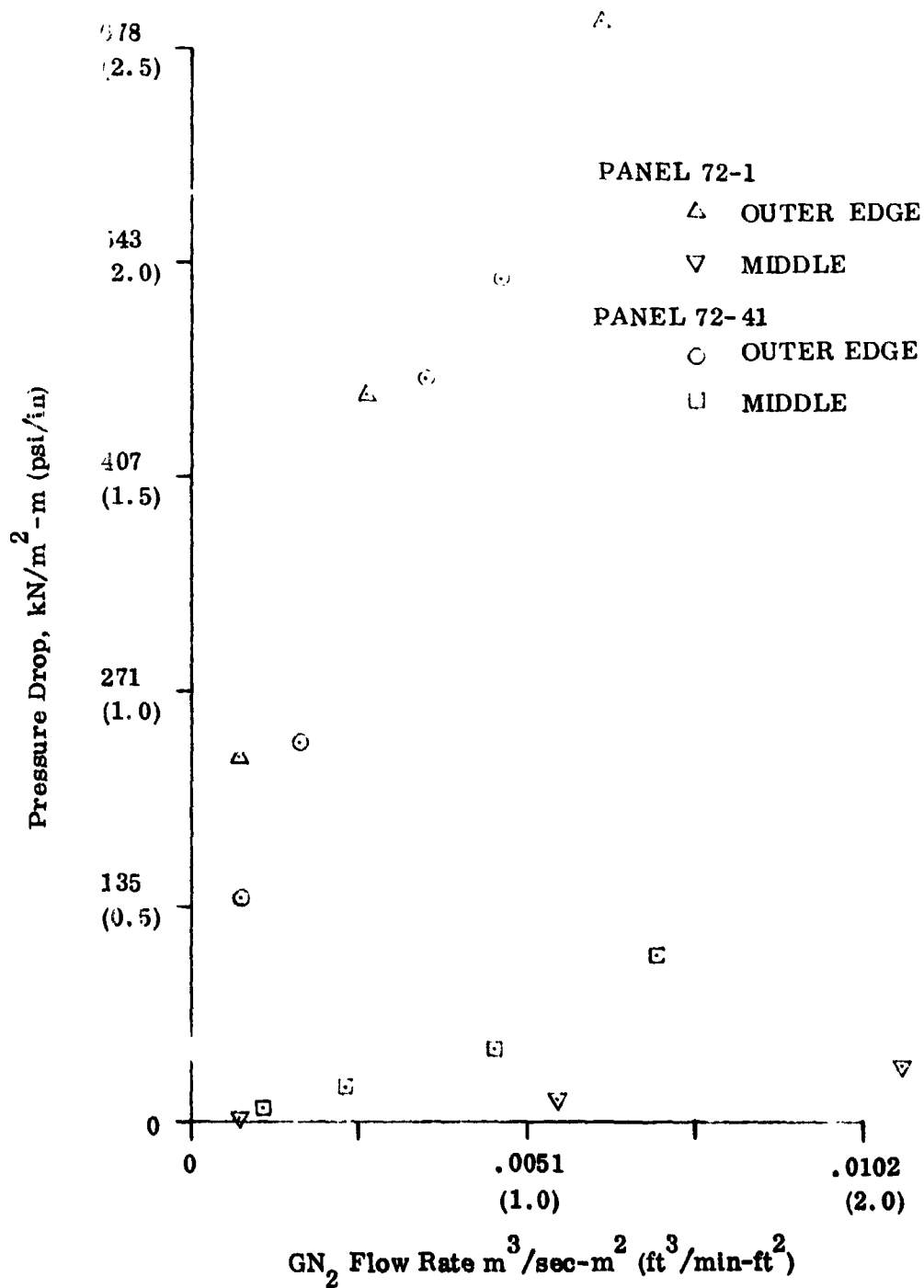


Figure 7-10. Variation of PPO Foam Permeability Perpendicular to Cells

Table 7-2. Thermal Performance Ranking, Composite (Vertical and Horizontal Orientations) Conductivity

<u>Panel</u>	<u>Rank</u>	Relative Conductivity
		<u>Ratio</u>
72-41	1	1.000
72-74	2	1.038
72-1	3	1.086
72-56	4	1.097
72-64	5	1.104
72-17	6	1.710

The X-ray inspection of the panels is most convenient in that it is non-destructive and can be performed as the panels are received before any machining is started. Sectioning panels after X-raying revealed that sometimes large voids are not evident in the X-rays. Also, undesirable cell curvature and low density center layers are not detectable by X-ray. Comparing X-rays of the panels in Table 7-2, the three highest ranked panels had a very uniform appearance. But, the sixth ranked panel also appeared quite uniform. Thus, if the X-ray reveals a defect, then the panel will likely have a poor thermal performance, but a panel with a uniform appearing X-ray might also have defects and a poor thermal performance.

Calculation of density variations throughout each panel and comparison with thermal performance reveals that panels with a more uniform density have a better thermal performance. Although there is no large difference in the uniformity of the three best thermal performance panels, Panel 72-41 had slightly smaller density variations than Panels 72-74 and 72-1, which were about equal in uniformity. Panels formed to their final thickness, such as Panels 72-56, -64 and -17 (the three poorest thermal performance panels), have significantly greater density variations than panels cut from the surface of a thick panel (Panels 72-41, -74 and -1). Thus, thermal performance is moderately sensitive to the degree of density variation.

Resistance to gas flow both perpendicular and parallel to the foam cells was measured. Ideally, the flow resistance should be infinite perpendicular to the cells (to prevent lateral convection) and near zero parallel to the cells. The results of the gas flow tests are listed in Table 7-3. Panels 72-18, -55 and -60 are similar to Panels 72-17, -56, and -64 (Table 7-2) in that they were originally formed as 25.4 mm (1.0 in.) panels. As seen from Table 7-3, the higher thermal performance panels have a higher gas flow resistance perpendicular to the cells. The low gas flow resistance in both directions of Panels 72-18, -55 and -60 is due to the low density center section of panels formed to their full final thickness. Such full thickness panels typically have a poor thermal performance, probably due to lateral convection through the porous

Table 7-3. Gas Flow Resistance

<u>Panel</u>	<u>GN₂ Gas Flow Resistance kN sec/m⁴</u> <u>(psi/in) (ft³/min-ft²)</u>		<u>Ratio</u>
	<u>Perpendicular</u>	<u>Parallel</u>	
72-41	3911 (0.0732)	1026 (0.0192)	3.8
72-74	6304 (0.118)	581 (0.0109)	10.8
72-1	4381 (0.0820)	1159 (0.0217)	3.8
72-18	1143 (0.0214)	310 (0.00580)	36.9
72-55	930 (0.0174)	104 (0.00195)	8.9
72-60	3489 (0.0653)	641 (0.0120)	5.4

midsection. Thus, high resistance to gas flow perpendicular to the foam cells is more important than low resistance parallel to the cells for good thermal performance.

Of the three techniques for predicting foam panel thermal performance, sectioning a panel for inspection and density gradient analysis is the best. A density gradient analysis will define porous regions, the same result obtained with the more difficult and time consuming gas flow resistance tests, and reveal defects not seen in X-rays. The X-ray technique is good in that it can be performed on all panels, but it has not been developed to the point where it can be relied upon to detect all panel defects.

SECTION 8

FOAM SURFACE RIGIDIZATION

Rigidizing the surface of a PPO foam internal insulation system exposed to liquid hydrogen reduces the potential for mechanical damage during ground and flight operations and reduces the amount of "debris," the partially broken cell wall fragments, attached at the surface of the foam, which may detach from the foam. The debris, which results from the cutting of PPO foam, is not due to any brittleness or lack of toughness inherent in the PPO polymer itself, but to the extreme thinness of the cell walls and the cutting techniques used. Elimination of the debris by hardening the foam surface must be achieved without sealing the open cells thus negating the advantages of the open-cell structure. The foam surface is hardened by thickening or strengthening the cell walls by either surface impregnation with additional polymer or by controlled surface shrinkage. Both of these methods have been investigated.

8.1 SURFACE IMPREGNATION

When an organic foam is exposed to a solvent, partial dissolution of the foam occurs. Due to surface tension, dissolution is accompanied by shrinkage and thickening of the cell edges. It is necessary to control this dissolution so closely that only the immediate surface of the foam is affected and to the exact extent required. The ends of the surface cell walls, which are on the order of $3\text{ }\mu\text{m}$ thick, must be significantly thickened without sealing off or otherwise compromising the essential, open-cell PPO foam structure.

Initially the effects of the following three solvents were investigated: carbon tetrachloride, chloroform, and methylene chloride, alone and in combination with a non-solvent, acetone. When any of these solvents was swabbed on the PPO foam surface, complete dissolution occurred. Similar results were obtained with solvent/non-solvent mixtures down to 10% solvent concentrations. At solvent concentrations of 10% or less (i.e. $\geq 90\%$ by volume of acetone), the foam was completely unaffected. In an attempt to more precisely control this dissolution process, various amounts of two different mixed solvents (consisting of 10% chloroform/90% hexane and 10% chloroform/90% methyl alcohol) were sprayed on the PPO foam surface via a glass atomizer. Microscopic examination disclosed that the chloroform/hexane mixed solvent weakened the PPO surface and increased the amount of "debris." By contrast, the chloroform/methyl alcohol mixed solvent decreased the amount of "debris" although the cell walls were not measurably thickened or the surface strengthened. Efforts to obtain further strengthening by increasing the chloroform content resulted in catastrophic dissolution of the foam.

Since it was extremely difficult to attain a satisfactory compromise between destructive dissolution on one hand and complete non-solvency on the other with the binary mixed solvents, an attempt was made to reduce the "chemical activity" of the system by dissolving varying amounts of PPO foam in the more promising mixed solvents. The presence of dissolved PPO resin, however, did not appear to significantly affect the solvent behavior of the mixed solvent. Subsequently, it was decided to investigate the interaction of PPO foam with pure benzene containing varying amounts of dissolved PPO resin. These experiments disclosed that any benzene solution, saturated or even supersaturated with respect to PPO resin, will attack and dissolve the PPO foam, even if the benzene solution must precipitate out a portion of the PPO resin which it already holds in a dissolved state. This is a direct consequence of the thermodynamic instability of a plastic foam relative to the bulk polymer. Due to its cellular nature, the foam is a higher free energy system.

Undoubtedly the PPO cell edges could be strengthened by depositing a different resin from a solution which is a non-solvent for the PPO foam. However, due to the large number of cryogenic cycles required of a Space Shuttle component, it seems unwise to attempt to strengthen the foam by coating it with an inherently incompatible polymer which might subsequently flake or peel off.

8.2 RADIANT HEATING

In contrast to the resin impregnation investigation described above, heat hardening of PPO foam was almost immediately successful. The initial experiments were performed using a 12.7 x 17.8 cm (5 x 7 in.) laboratory infrared heater containing five 500 watt, General Electric T3 quartz lamps. These preliminary experiments confirmed past experience that the best results were obtained at high radiant fluxes for short time periods. Accordingly, two additional lamps were added to the heater, bringing it to its maximum capacity. Since each lamp has an effective radiating length of 12.7 cm (5 in.), the radiant heat flux density is 217 kW/m² (140 W/in²). PPO foam specimens, 7.6 x 10.1 x 1.8 cm (3 x 4 x 0.7 in.), were centered at various distances below the quartz lamps and irradiated for various lengths of time. After cooling to room temperature, the thickness of the specimen was measured at five positions across the surface, the hardness was checked qualitatively by scraping with a fingernail, and finally the specimen examined at from 10X to 30X magnification by both transmitted and incident illumination under a stereomicroscope.

The results obtained with an early specimen were informative. The specimen was placed at an 0.35 rad (20°) slant under the infrared heater operating at full power (110 V.) for 5 seconds. Since one end of the foam was closer to the heater, a heat gradient was applied to the specimen. As shown in Figure 8-1, the sample was badly warped. In general, the edges do not shrink and, therefore, the remaining 90% of the surface is "dished" with the low point toward the center. Most specimens placed horizontally (i.e. parallel to the heater lamps) show this general type shrinkage.

In the slanted orientation, the upper, hotter end of the specimen sagged. Moreover, this upper end was burned black while the lowest, coolest portion was virtually unchanged. The upper 50% of the surface was definitely hardened. Microscopic examination disclosed that the lower half still had the usual debris, but starting with the brown portion of the surface approximately half-way up, the debris had been melted back into the cell walls. This melted debris plus the 10% shrinkage had thickened the upper cell walls approximately six-fold without closing off the 0.01 to 0.03 in. diameter cells. Stereoscopic examination with transmitted light verified that the lower portions of the foam cells were unaffected by this infrared heating. In general, the open PPO foam cell structure was not significantly compromised until the 50% volume shrinkage-point was reached. Even the black pyrolyzed areas still retained a high proportion of open cells.

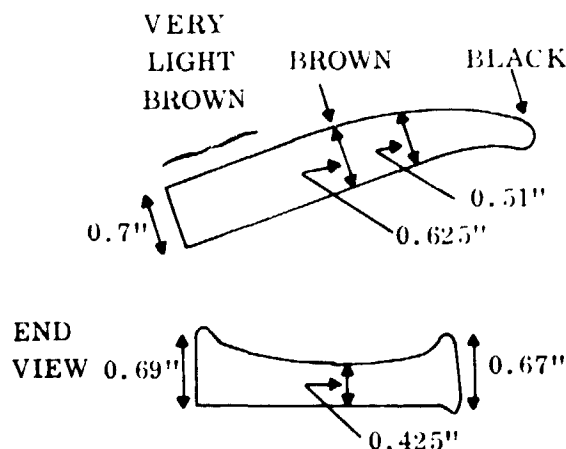


Figure 8-1. Results of Slanted Specimen Test

Radiant heating produced surface hardening and melted surface debris without closing the PPO foam cells. Radiant heating also tended to produce an irregular surface. The heat hardening process is time and distance dependent, therefore a series of experiments were performed to minimize surface irregularities and select the proper time and distance. Results of these experiments are summarized in Table 8-1. These results were checked in four instances and found to be reproducible. The most uniformly hardened surfaces were obtained in experiments 14 and 15 in which the specimens were exposed to the full 217 kW/m^2 (140 W/in^2) illumination for 4 seconds at a distance of 1.9 cm (0.75 in). Unfortunately, in runs 14 and 15, as in all other experiments, the heat-hardened specimens exhibited characteristic ridges around the entire upper edges plus a concave center section. Since specimens ranging in size from $2.5 \times$ to $7.6 \times 10.2 \text{ cm}$ (1×1 to $3 \times 4 \text{ in.}$) show this same ridge/concave center whether run in the small laboratory infrared heater or in a $0.91 \times 1.22 \text{ m}$ ($3 \times 4 \text{ ft}$) factory heater, it appears to be an edge effect associated with the radiation absorption/reradiation balance of the PPO foam specimen itself. Neither the peripheral rim or the concavity of the center is affected by the reflectivity of the base upon which the radiant heating is performed. However, by wrapping a foil guard-barrier 3.8 cm (1.5 in.) high around the edge of the specimen, the peripheral rim was sharply diminished and the central concavity eliminated. The cross section of a typical 1.8 cm (0.7 in.) thick PPO foam specimen (#23) hardened by irradiation for 4 seconds

Table 8-1. Effect of Time/Distance on the Infrared Heating of PPO Foam

Conditions:			
12.7 × 17.8 cm (5 × 7 in.) I.R. Heater Operating at 110 V; 217 kW/m ² (140 W/in ²)			
Specimen No.	Distance From Lamp cm (in.)	Irradiation Time sec	Results
2	6.4 (2.5)	5	No shrinkage. No detectable hardening. Debris only partially removed.
3 and 8	6.4 (2.5)	10	Uneven shrinkage to 5.1 mm (0.2 in.). Surface hardened. Debris eliminated.
4	3.2 (1.25)	5	No shrinkage. No hardening. Debris unchanged.
5 and 13	1.9 (0.75)	5	Uneven shrinkage 1.8 to 3.6 mm (0.07 to 0.14 in.). Surface hardened. Debris eliminated.
9	2.5 (1.0)	2	No shrinkage. No hardening. Debris unchanged.
10	2.5 (1.0)	4	No shrinkage and no hardening. Debris removed.
11 and 12	2.5 (1.0)	5	4.1 mm (0.16 in.) shrinkage, max. (at center). Debris removed, surface hardened.
14 and 15	1.9 (0.75)	4	2.3 mm (0.09 in.) shrinkage, max. Debris removed, surface hardened.

at 217 kW/m² flux and a lamp-to-specimen distance of 1.9 cm, varied from 1.70 to 1.71 cm (0.670 to 0.675 in.) at the edges to 1.60 to 1.63 cm (0.630 to 0.640 in.) in the center. The effect of guard-barrier height on surface uniformity was not quantitatively determined. However, from the factory scale-up discussed below, the optimum height would appear to be between 5.1 and 7.6 cm (2 and 3 in.).

All of the preceding experiments were performed with the infrared heater operating at full voltage. As already stated, this amounts to a radiant heat flux of 217 kW/m². Since most commercial infrared heaters have a maximum output of approximately 115 kW/m², tooling and operating costs for rigidizing full size panels could be minimized if uniform heat-hardening could be accomplished with lower power outputs over longer exposure times. The effect of using lower infrared

radiation fluxes and longer exposure times on the standard laboratory-size PPO foam specimens is summarized in Table 8-2. Here it was apparent that the 4 sec 217 kW/m^2 (140 W/in^2) heating cycle still produced the most uniformly hardened foam. Fairly uniform, hardened foam, however, could be obtained by running the infrared heaters at 55 volts rather than 110 volts. With a calculated flux of 56 kW/m^2 (36 W/in^2) it appears that an exposure time of about 11 or 12 seconds would be optimum. The lowest flux, however, was definitely insufficient to uniformly harden the PPO foam. Judging from the results, it is probable that any infrared heater with a power output greater than 25 kW/m^2 (16 W/in^2) could be used to heat-harden PPO foam.

In view of these promising laboratory results, the thermal hardening process was applied to full-size PPO foam panels, $63.5 \times 73.7 \text{ cm}$ ($25 \times 29 \text{ in.}$). A large, 290 V/45A industrial infrared heater was used. This heater consisted of an assembly of 25.4 and 40.6 cm (10 and 16 in.) quartz lamps arranged 1.3 to 4.4 cm (0.5 to 1.75 in.) apart, contained in a $1.1 \times 0.9 \text{ m}$ ($42 \times 37 \text{ in.}$) frame. This heater was mounted, somewhat off-center, over a female die 61 cm (2 ft) in diameter. This female die was used as a convenient base on which the PPO foam panels rested during the irradiation. Three foam panels were heat hardened at the maximum power setting. In all cases, 7.6 cm wide \times 76 μm thick (3 in. wide \times 3 mil thick) stainless steel foil was used as a guard-barrier around the edge of each panel. Results for the three panels are summarized in Table 8-3, arranged in the order in which they were run. Figure 8-2 shows the last panel after surface hardening and Figure 8-3 shows a cross-section through the edge of this panel. Since only one of four ohmmeters was working correctly (registering 290V at 45 amps.), the power output could unfortunately not be calculated.

The irradiation times of 12-15 seconds were selected on the basis of the behavior of small $17.8 \times 5.1 \times 2.5 \text{ cm}$ ($7 \times 2 \times 1 \text{ in.}$) blocks of PPO foam in three preliminary runs. It seems probable that irradiation times of 16-20 seconds would have produced a higher degree of hardening without loss of surface uniformity. If non-uniformities do arise, it is believed they can be suppressed by rotating the PPO foam panel during irradiation. If this is inconvenient, merely running the irradiation in two equal stages and rotating the foam panel 90° between the stages should improve the surface hardness uniformity. Based on these pilot experiments, heat hardening PPO foam panels appear to have merit as a method for providing surface protection.

8.3 QUANTITATIVE EVALUATION OF HEAT HARDENED PPO FOAM

Since large changes in surface hardness and debris were required, it was most convenient to monitor the progress in hardening by simple qualitative tests, such as scratching with a fingernail and microscopic examination. However, once an effective, economical procedure for rigidizing the foam was developed, it was appropriate to develop a more precise quantitative measure of the improvement achieved.

The rationale behind this investigation was that hardening the surface would make it less susceptible to mechanical damage and would minimize any sloughing off of

Table 8-2. Effect of Time/Voltage Variations on the Heat Hardening of PPO Foam

Conditions:					
12.7 × 17.3 cm (5 × 7 in.) I. R. Heater Containing 7500 G.E. T-3 Quartz Lamps			1.9 cm (0.75 in.) Specimen-to-Lamp Distance		
7.6 × 10.1 × 1.8 cm (3 × 4 × 0.7 in.) Specimen			3.8 cm (1.5 in.) Guard-Barrier Height		
Experiment Number	Applied Voltage	Calculated Flux kW/m ² (W/in. ²)		Time Sec	Results
M97-22	110	217	(140)	2	Debris diminished slightly. Negligible hardening and thickening of cell wall. No rim.
M97-20	110	217	(140)	3	Debris removed. Minor hardening and thickening of cell walls. Negligible rim.
M89-30	110	217	(140)	4	Debris removed. Surface hardened. Cell walls thickened. Small rim 0.76 mm (0.03 in.) high.
M97-27	55	56	(36)	10	Debris removed. Surface hardened and cell walls thickened but less than at 4 sec/217 kW/m ² above.
M97-32	55	56	(36)	16	Excessive heating. Surface melted and slumped. Up to 40% shrinkage. Rim on 3 sides only.
M98-14	28	14	(9)	30	Debris slightly removed. No hardening or cell thickening
M98-16	28	14	(9)	45	Debris removed but negligible hardening and cell thickening.
M98-19	28	14	(9)	45 + 30	Debris removed. Some hardening and cell thickening but surface concave.
M98-3	28	14	(9)	60	Debris removed. Surface hardened and cells thickened about the same as 10 sec/55 V experiment (M97-27) above. Surface much more uneven and highly concave.

Table 8-3. Surface Hardening Full-Sized Panels

Panel Dimension cm (in.)	Heater To Panel Distance cm (in.)	Irradi- ation Time	Results
63.5 × 73.7 × 1.8 (25 × 29 × 0.7)	21.1 (8.3)	12 Sec	Fairly uniform browning; debris partially removed. Minor hardening and cell wall thickening. No peripheral ridges.
63.5 × 73.7 × 2.5 (25 × 29 × 1.0)	22.9 (9.0)	12 Sec +5 Sec	Quite uniform. Debris removed. Some hardening and cell wall thickening. No peripheral ridges.
63.5 × 73.7 × 2.5 (25 × 29 × 1.0)	22.9 (9.0)	15 Sec	Uniform browning. Debris removed. Moderately hardened surface with thickened cell walls. Edges somewhat melted rather than raised.

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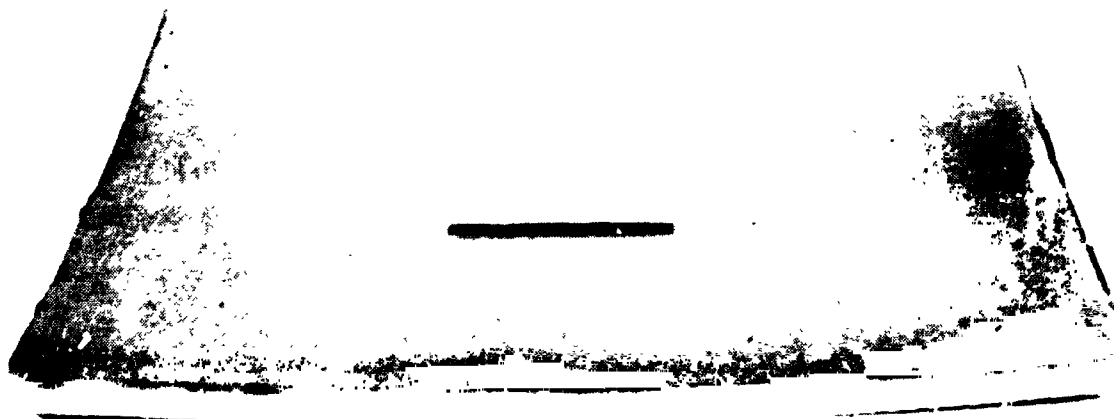


Figure 8-2. PPO Foam Panel Surface Hardened by Infrared Heater, 63.5 × 73.7 cm (25 × 29 in.)



Figure 8-3. Cross-section Through Edge of Surface Hardened Foam Panel

friable cell fragments (which might clog filters, contaminate the working fluid, etc.) under actual service conditions. Two different tests were used. The first was an abrasion test based on a standard, commercial instrument called the Tabor Abrader. This instrument contains two weighted, balanced, grinding wheels which rotate freely against a 10.2 cm (4-in.) diameter specimen, which in turn is mounted on a motor driven turntable. The weight loss suffered by the plastic specimen after a predetermined number of rotations is taken in Federal Test Method Standard No. 406 as a measure of abrasion resistance. Like any other abrasion tester, the Tabor Abrader cannot serve as a precise tool for accurately predicting abrasion service life. This is particularly true in the present case where the handling conditions experienced by the hardened foam do not resemble in any way "drag" against a grinding wheel.

The second test is completely non-standard, but is considered to be a valid quantitative measure of the tendency of the foam to slough off particulate matter. In this latter test, standard size plugs of both virgin and hardened PPO foam were suspended in an "inert" fluid and subjected to ultrasonic vibration for a given time. The amount of particulate matter sloughed off by the various foam plugs was determined by a particle analysis of the suspending fluid. In all these quantitative tests, virgin PPO foam specimens were compared with specimens which have been heat hardened under the best known conditions, i. e. 4 sec at 217 kW/m^2 (140 W/in^2), 1.9 cm (0.75 in.) from lamps. The abrasion tests were performed at three load ranges: 250, 500, and 1,000 g. (.55, 1.1, 2.2 lb. The weight losses

suffered both virgin and heat hardened PPO foam specimens are shown in Tables 8-4, 8-5, and 8-6 for these three loads, respectively. Under the 250 g (.55 lb) load both the virgin and heat hardened PPO foams are abraded at about the same rate. Under the two higher loads, however, the heat hardened foams consistently show lower abrasion rates than the virgin foam. Figure 8-4 illustrates the results of the abrasion tests using the 1000 g (2.2 lb) load. It can be seen that the hardened surface resists the abrasion until it is removed at about 300 cycles.

The ultrasonic evaluation for sloughing was performed with 3.3 cm (1.3 in.) diameter plugs cut from heat hardened and virgin foam. The walls and back surface of these plugs were sealed by coating them with a clear polyurethane paint, Desothane. Only the subject surfaces were left unsealed. The plugs were then suspended in 50 ml (3.1 in³) beakers, subject surface down. Sufficient methanol was introduced to submerge the subject surface, and the assembly was subjected to 4 minutes vibration in a laboratory "Sonoblaster" ultrasonic bath. The methanol solutions were then analyzed for particles according to ASTM F3-12. The results, Figure 8-5, indicate that the number of particles sloughed off by the virgin specimen is 3 to 5 times greater than that for the heat hardened specimen.

Table 8-4. Weight Loss Suffered by Virgin and Heat Hardened PPO Foam Specimens on the Tabor Abrader

Condition: 250 g load

Specimen Number	0 Abrader Cycles		50 Abrader Cycles		100 Abrader Cycles		200 Abrader Cycles		300 Abrader Cycles	
	Spec Wt	% Orig Wt	Spec Wt	% Orig Wt	Spec Wt	% Orig Wt	Spec Wt	% Orig Wt	Spec Wt	% Orig Wt
<u>Virgin</u>										
1	2.1384g	100%	2.1173g	99.0%	2.0858g	97.5%	2.0247	94.7%	1.9655g	91.9%
2	2.1071	100%	2.0862	99.0%	2.0562	97.6%	1.9963	94.7%	1.9382	92.0%
3	2.1963	100%	2.1683	98.7%	2.1335	97.1%	2.0646	94.0%	1.9974	91.0%
Average		100%		98.9%		97.4%		94.45%		91.6%
<u>Heat Hardened: 217 kW/m² (140 W/in²)</u>										
1	2.3151g	100%	2.2804g	98.7%	2.2468g	97.0%	2.1758g	94.0%	2.1072g	91.0%
2	2.3479	100%	2.3179	98.7%	2.2853	97.3%	2.2185	94.5%	2.1525	91.7%
3	2.1111	100%	2.0901	99.0%	2.0616	97.7%	2.0064	95.0%	1.9534	92.5%
Average		100%		98.8%		97.3%		94.5%		91.8%

Table 8-5. Weight Loss Suffered by Virgin and Heat Hardened PPO Foam Specimens on the Tabor Abrader

Condition: 500 g load

Specimen Number	0 Abrader Cycles		50 Abrader Cycles		100 Abrader Cycles		200 Abrader Cycles		300 Abrader Cycles	
	Spec Wt	% Orig Wt	Spec Wt	% Orig Wt	Spec Wt	% Orig Wt	Spec Wt	% Orig Wt	Spec Wt	% Orig Wt
<u>Virgin</u>										
1	2.2653g	100.0%	2.2213g	98.0%	2.1452g	94.8%	2.0577g	90.7%	1.9740g	87.2%
2	1.8874	100.0%	1.8412	97.6%	1.7875	94.7%	1.6952	89.9%	1.6132	85.5%
3	2.1133	100.0%	2.0679	97.9%	2.0176	95.5%	1.9261	91.1%	1.8419	87.2%
4	1.8479	100.0%	1.8059	97.7%	1.7606	95.3%	1.6745	90.6%	1.5971	86.4%
Average		100.0%		97.8%		95.1%		90.6%		86.6%
<u>Heat Hardened; 4 sec/217 kW/m² (140 W/in²)</u>										
1	2.2307g	100.0%	2.1928g	98.4%	2.1606g	97.0%	2.0805g	93.4%	2.0015g	90.0%
2	2.1584	100.0%	2.1204	98.4%	2.0756	96.1%	1.9876	92.1%	1.9007	88.1%
3	2.0078	100.0%	1.9651	97.9%	1.9184	95.5%	1.8333	91.3%	1.7566	87.5%
4	1.9143	100.0%	1.8748	97.9%	1.8351	95.9%	1.7635	92.1%	1.6969	88.6%
Average		100.0%		98.15%		96.1%		92.25%		88.55%

Table 8-6. Weight Loss Suffered by Virgin and Heat Hardened PPO Foam Specimens on the Tabor Abrader

Condition: 1000 g load

Specimen Number	0 Abrader Cycles		50 Abrader Cycles		100 Abrader Cycles		200 Abrader Cycles		300 Abrader Cycles	
	Spec Wt	% Orig Wt	Spec Wt	% Orig Wt	Spec Wt	% Orig Wt	Spec Wt	% Orig Wt	Spec Wt	% Orig Wt
<u>Virgin</u>										
1	2.2953g	100.0%	2.1697g	94.5%	2.0580g	89.7%	1.8721g	81.6%	(1)	-
2	1.1094	100.0%	2.0035	95.0%	1.9022	90.2%	(2)	-	-	-
3	2.2483	100.0%	2.1499	95.6%	2.0649	91.9%	1.8834	83.8%	1.7480	77.7%
Average		100.0%		95.0%		90.6%		82.7%		77.7%
<u>Heat Hardened; 217 kW/m² (140 W/in²)</u>										
1	2.1501	100.0%	2.0514g	95.4%	1.9584g	91.1%	(3)	-	-	-
2	2.2074	100.0%	2.1091	95.5%	2.0222	91.6%	(4)	-	-	-
3	2.2423	100.0%	2.1665	96.6%	2.0872	93.1%	1.9237g	85.8%	1.7734g	79.1%
Average		100.0%		95.8%		91.9%		85.8%		79.1%

(1) Specimen tore apart at 230 cycles.

(2) Specimen tore apart at 170 cycles.

(3) Specimen tore apart at 138 cycles.

(4) Specimen tore apart at 174 cycles.

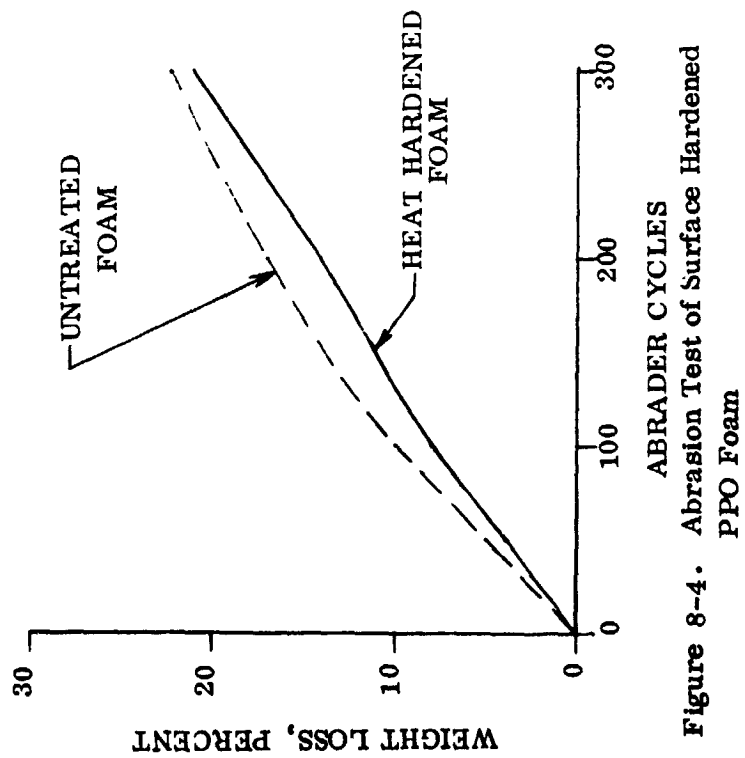


Figure 8-4. Abrasion Test of Surface Hardened PPO Foam

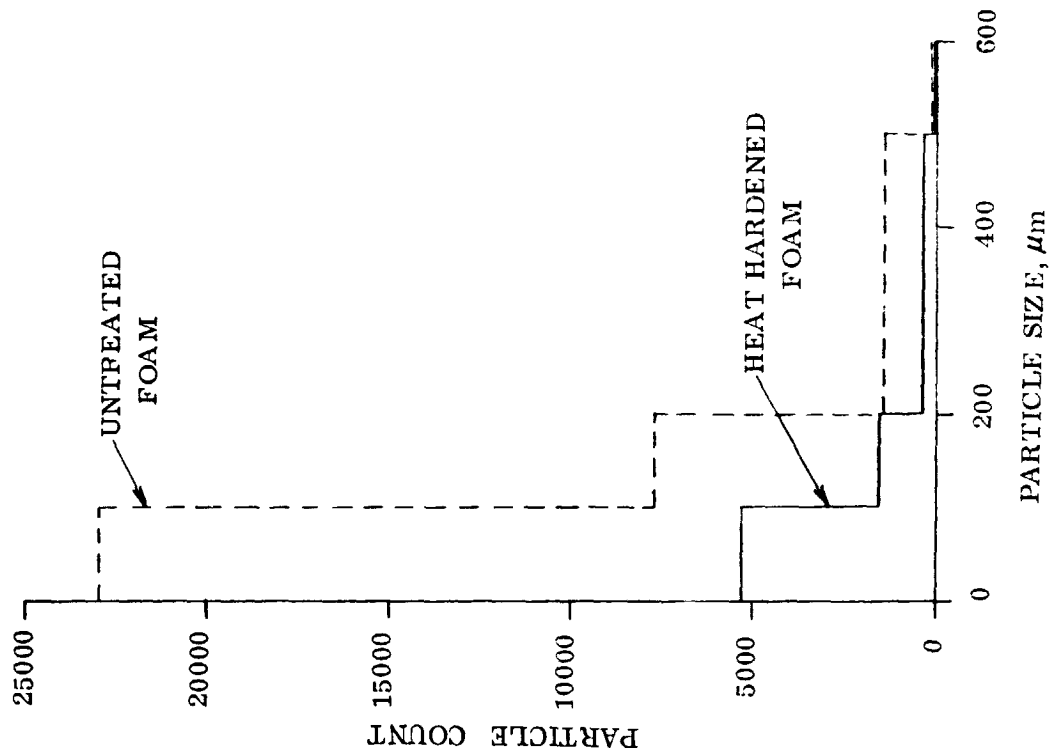


Figure 8-5. Particulate Sloughing of PPO Foam

SECTION 9

PANEL JOINTS AND REPAIRS

PPO foam is currently fabricated in panel sizes less than one meter square. The anticipated size of production panels probably will not exceed two meters. Thus any large tank will contain numerous panel-to-panel joints as well as closeouts. Because of the extremely high elasticity of PPO foam in the direction perpendicular to the cells, unbonded compressed joints are both attractive and feasible. Prebonding the smaller panels into the larger sheets may be desirable to reduce final assembly costs and on-station time. Edge bonding during single panel installations may be desirable or even mandatory in hardpoint closeout areas. Whenever a repair is made by removing and replacing a panel or portion thereof, a joint will occur. In this case the ability to compress and insert a repair panel is one of the most redeeming features of PPO foam. The repair of any bonded system necessitating adhesive removal is difficult; PPO foam repair is no exception. Repairs however, were made and successfully tested in LH_2 .

9.1 PANEL JOINTS

Various methods of joining PPO foam panels have been considered. Since the available foam panels are approximately one meter square, many panels will have to be jointed together to insulate an LH_2 tank. During normal operation of the tankage system, the inner surface of the insulation sees liquid hydrogen temperature (21K (37R)) while the tank surface remains relatively warm, i.e. 200K (360R) to room temperature. At this time, the tank structure experiences maximum strains as a result of pressurizing while the inner surface of the insulation undergoes thermal contractions of approximately one percent. To maintain a reliable joint between panels, either an adhesive bonded joint is required or one providing sufficient residual edge compression to account for the tendency to gap.

The panel joints must not inhibit the insulation by closing off foam cells or by creating heat shorts to the tank skin. Due to the very large number of panel joints involved, the problem of heat shorts could be quite severe. The panel joints must be capable of withstanding the structural and thermal stresses involved in an LH_2 tank and be structurally compatible with the foam (no excessive thermal stresses between the joint and the foam). Also, the panel joints must allow the use of practical assembly techniques.

Tongue-and-groove joints and other types of lap joints would involve closing off foam cells, especially if the joints were to be bonded. Closed cells would be subject to pressure cycling and eventual failure. Without bonding the joint, there would be

nothing to restrain the lip of a lap joint from pulling up from the surface, leaving gaps and voids in the joint. Bonding a lap joint would create large angular areas of adhesives in the foam which could result in severe thermal stress problems. Finally, since the only requirement of the panel joints is to provide continuity of the foam insulation, the strength of the joint, aside from its own structural integrity, is unimportant, negating any possible structural advantage of a lap joint.

Butt joining the PPO foam panels is very simple and is most compatible with the open-cell insulation system in that a butt joint does not close off the foam cells. The bonded butt joint is a positive joint which can be made during the original installation of the foam without special tooling. Adhesive on a foam butt joint will make the final assembly more difficult at close out panel installation and would be difficult to use with a repair plug since the foam, as it is pressed into place, will tend to scrape the adhesive from the sidewalls and into the bottom of the joint area. The surrounding foam areas would have to be masked to protect them from adhesive that would be scraped off the edge of the panel being installed. In addition, the bonded butt joint results in a hardpoint discontinuity and associated structural and thermal stresses in the adhesive layer and adjacent foam and presents a heat short through the foam insulation.

PPO foam panels lend themselves to the use of compressive butt joints. The material's low modulus and good ductility in the direction perpendicular to the foam cells allows the panels to be compressed by up to 15 percent without any damage and with good recovery characteristics under room temperature conditions. The compression butt joint offers an easy installation method because adhesive is required only at the face adjacent to the tank wall. This method also avoids any possible heat shorts and structural and thermal stresses in the joint. The unbonded compressive butt joint has been successfully employed in a PPO foam insulation system for an LH₂ tank (Ref 3). The installation method was to first install alternate foam panels. After the tank wall bond for these panels had cured, the remaining panels were compressed and bonded into their spaces. Before the bond had cured on the remaining panels, the compression tooling was removed allowing the panels to expand into place, compressing the previously installed panel edges. A 2 percent residual edge compression was successfully utilized and tested in this tank system. The contraction of PPO foam from room temperature to 21K (37R) is about one percent. This installation in the 1000 gallon tank is shown in Figure 9-1.

9.2 PANEL REPAIRS

The need for the capability to repair PPO foam insulation that has been damaged after installation onto a tank skin becomes apparent when one considers the large insulated areas subject to accidental damage during fabrication and inspection. Also, possible damage due to an area that becomes unbonded as a result of repeated flight cycles or an area that was not properly bonded during installation might need repair. Any repair techniques used must meet certain general requirements. As

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Figure 9-1. PPO Foam Installed in 1000 Gallon, 53/4 Foot Diameter LH₂ Tank.

PPO foam in an internal installation, very low debris generation during repair is necessary in order to keep contamination of the tank to a minimum. Tools for repairing the insulation must be designed and used to minimize potential damage to the aluminum tank skin. The repair procedure must be capable of being performed on an overhead surface since it may not be possible to orient the tank so as to have the repair surface at the bottom. Two techniques for removing damaged foam were developed. Each used a cylindrical blade to first slice the foam around the repair area. Then either a hot wire or a phenolic cutter was used to remove the foam within the cylindrical blade. After removal of the damaged foam, abrasive disks were used to clean away the adhesive from the aluminum surface. In order to hold down the bonded repair during cure, a vacuum bagging technique was used. The use of these tools and techniques is detailed in this section.

9.2.1 FOAM REMOVAL. The first step in a panel repair is to apply a sheet of adhesive back mylar to the PPO foam surface to be repaired. The mylar sheet is essential to the vacuum bagging process used at the end of the panel repair procedure and also serves to protect the foam surface around the repair area. The mylar sheet

used in this development was 45.7 cm (18 in) wide and 50.8 μ m (0.002 in) thick. Then a cylindrical steel blade is placed around the repair area and inserted into the foam. The cylindrical blade, shown in Figure 9-2, is 15.2 cm (6.00 in) in diameter and the blade is 0.635 mm (0.025 in) thick. The depth of the blade, 4.45 cm (1.75 in), is such that when fully inserted, it does not completely penetrate the 4.57 cm (1.80 in) thick foam, thus preventing the steel edge from contacting the aluminum tank skin underlying the foam. Since it is not necessary for the cylindrical cutter to penetrate to the tank skin, this cutter can also be used on curve insulated surfaces. Figure 9-3 shows the cylindrical blade inserted in a PPO foam panel with the mylar sheet applied to the foam surface. The mylar has been peeled away at the repair area inside the cylindrical blade. The cylindrical blade remains in the foam panel to protect the foam adjacent to the repair until a new piece of foam is bonded into the repair area.

The first technique used to remove the damaged foam inside the cylindrical cutter was a hot wire. The hot wire will not damage the aluminum tank skin and this cutting method produces no debris, thus avoiding the added complication of a debris collecting system. Also, the hot wire cutter is easy to use even in an overhead repair and will easily remove foam from any tank contour. The hot wire cutting tool developed for this application is shown in Figure 9-4. The rectangular wire loop is 2.5 cm (1.0 in) wide by 5.1 cm (2.0 in) long and is made of 1.45 mm (0.057) diameter Nichrome wire. The phenolic insulating handle has two copper sockets with set screws for holding the wire loop. For cutting PPO foam, it was found that a wire temperature of 750K to 840K (900F to 950F) worked best. Figure 9-5 shows a repair hole in a PPO foam panel that has been partly cleared out using the hot wire cutting tool. Note how the cylindrical blade, inserted into the foam panel prior to removal of the damaged foam, protects the foam material adjacent to the hole. Also shown is a piece of foam removed by the hot wire.

The second technique used to remove the damaged foam inside the cylindrical cutter was by means of a phenolic cutting tool driven by an air motor. The designs for the cutter are shown in Figure 9-6. Phenolic was used for the cutter material to prevent any possible damage to the aluminum tank skin. The smaller diameter mill cutter (1.27 cm (0.5 in) diameter) was found to be too easily broken during use, but the 2.54 cm (1.0 in) diameter mill cutter proved to be satisfactory and also removed the foam material at a higher rate. The third cutter was a flat, 3.18 mm (0.125 in) thick, 3.81 cm (1.50 in) wide phenolic blade clamped into an aluminum arbor. The flat blade cutter proved to be the most satisfactory. The flat blade is much simpler to make than the mill cutter and it removes the foam at a higher rate. Also, the flat blade produced cutting debris that was easier to contain. The mill cutter tended to grind the PPO foam into dust-like particles whereas the flat blade shreaded the foam into thin pieces. Figure 9-7 shows the flat blade, phenolic cutter mounted on the air motor. The large (30 cm (12 in) diameter), circular face plate is covered with teflon and fits flush against the rim of the cylindrical blade in order to contain the cutting debris. The debris was collected by vacuum through the port located near the base of the cutting tool. Figure 9-8 shows the air motor riding on the rim of the cylindrical blade which



Figure 9-2. Cylindrical Blade for Cutting Around the Repair Area

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Figure 9-3. Cylindrical Blade Inserted in a PPO Panel

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Figure 9-4. Hot Wire Cutting Tool

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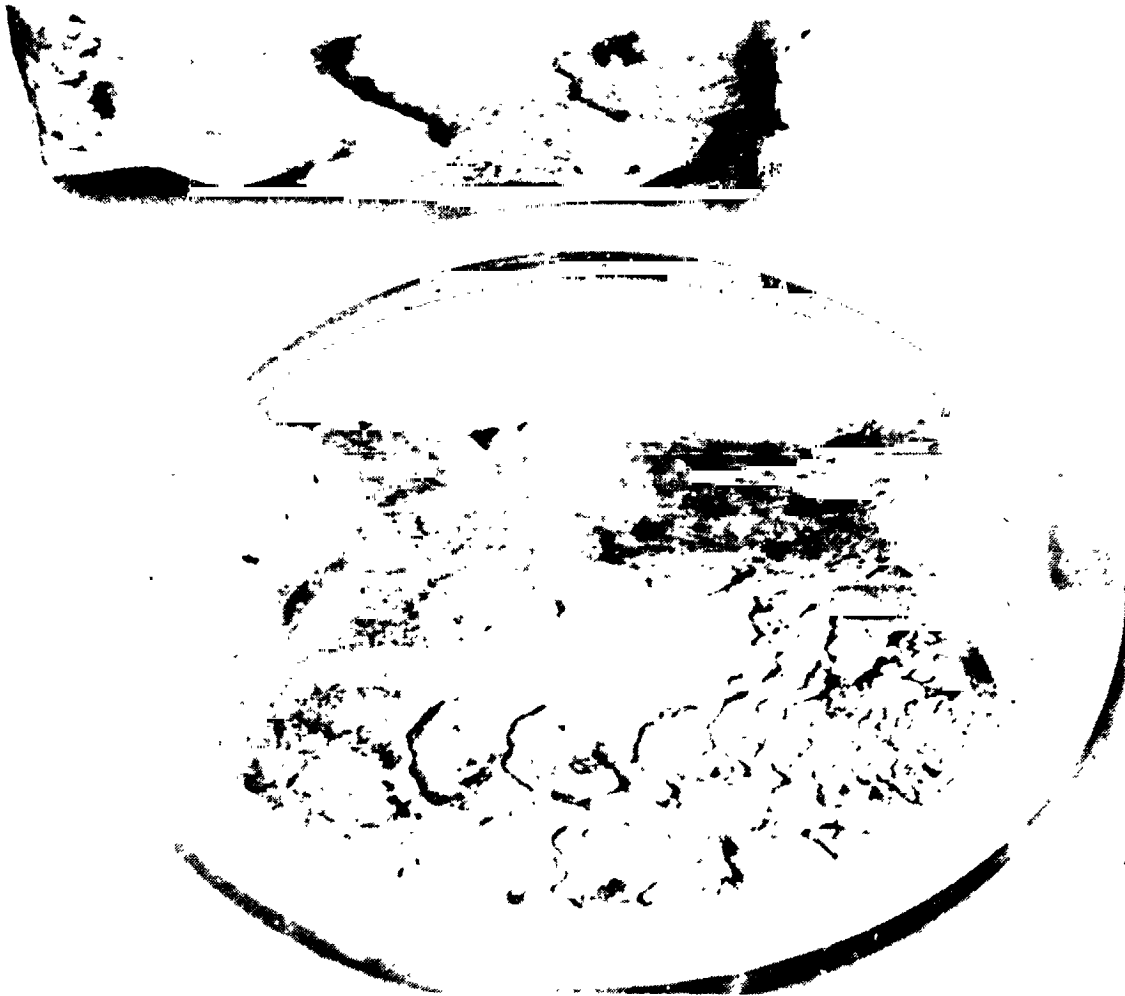
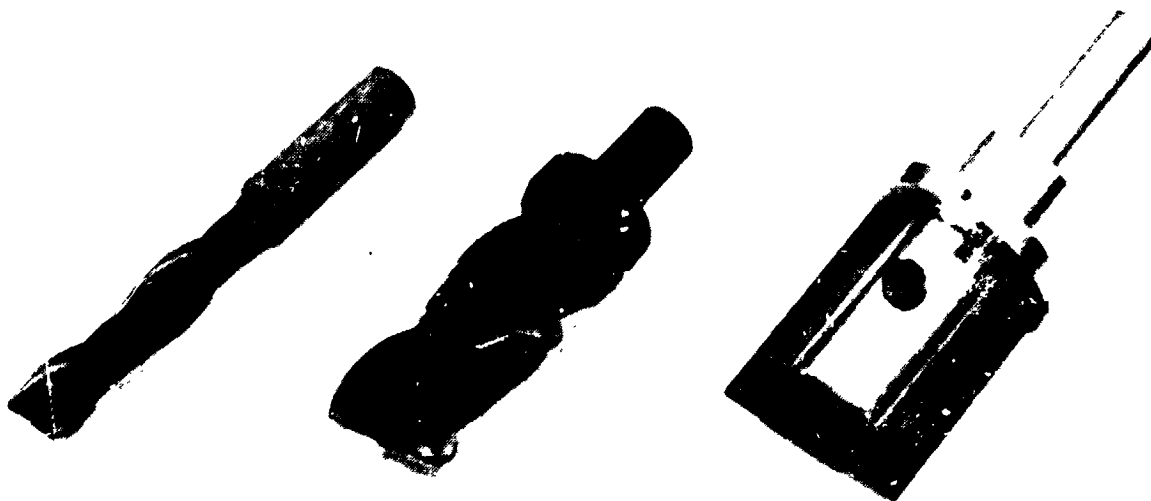


Figure 9-5. PPO Foam Removed Using the Hot Wire Tool

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Figure 9-6. Phenolic Cutting Tools

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Figure 9-7. Flat Blade Phenolic Cutter Mounted on an Air Motor With a Teflon Face Plate

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Figure 9-8. Air Motor Riding on Rim of Cylindrical Blade with Air and Vacuum Lines Attached

has been inserted into a PPO foam panel. The air supply to the motor and the vacuum hose can also be seen. In order to use the phenolic cutter without allowing the escape of any debris, it is necessary to first cut a starting hole by some hand method such as the hot wire so that the face plate is against the rim of the cylindrical blade before the air motor is started. Figure 9-9 shows a repair hole in a PPO foam panel that has been cleared out using the flat blade, phenolic cutter.

The preferred method for removing damaged PPO foam is the hot wire cutter. The hot wire cutter does not have the debris problem that the phenolic cutter has and thus does not require a vacuum system. The hot wire tool is much easier to handle than the air motor driven phenolic cutter. Finally, the hot wire technique could be used to remove an entire PPO foam panel whereas the phenolic cutter system would scatter large quantities of foam debris creating a contamination problem inside a tank.

9.2.2 ADHESIVE REMOVAL. The most difficult job in the repair procedure is the adhesive removal. There are three potential methods:

- Cut the adhesive around the periphery of the foam cut-out and scrape out the repair area. This is the most desirable but is difficult or even impossible depending on bond quality and adhesive flexibility.

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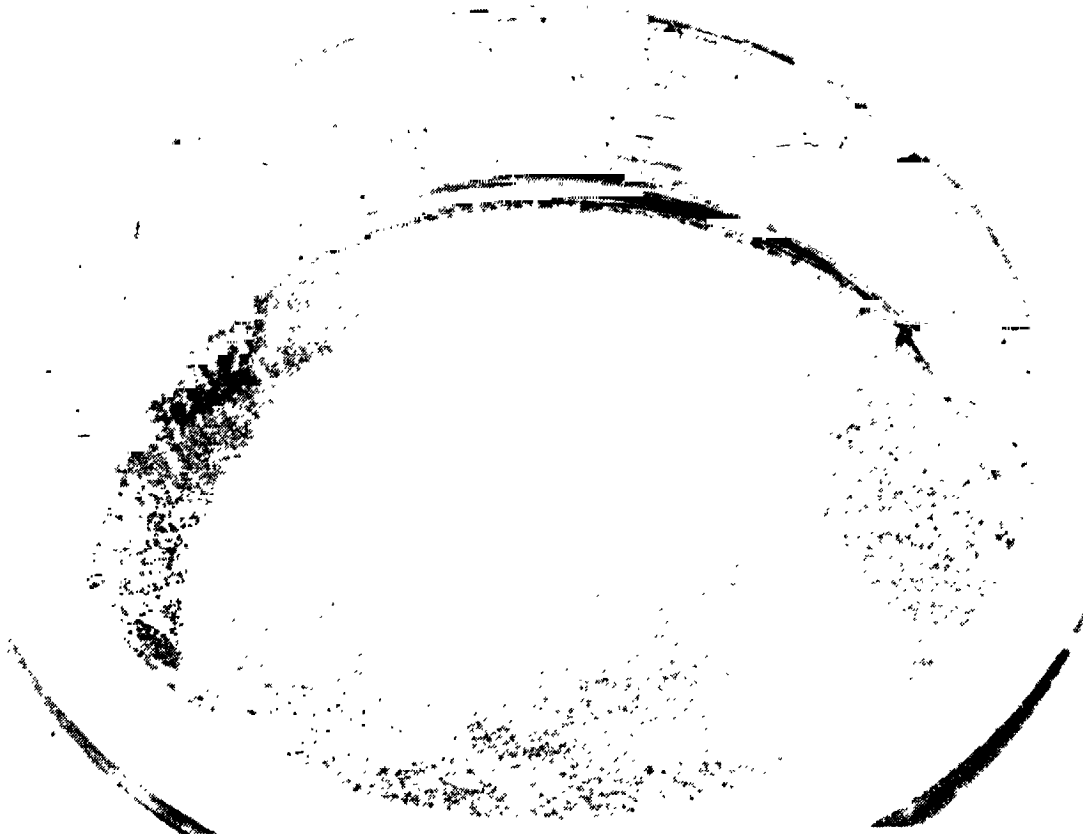


Figure 9-9. Repair Hole Cleared Out Using the Flat Blade, Phenolic Cutter

- Dissolve or soften the adhesive with a solvent then scrape it out.
- Mechanically grind the adhesive out with a hard plastic cutter or abrasive disc.

In all cases the difficulty lies in preventing damage to the repair area periphery and in avoiding solvent and debris contamination in the tank and insulation.

An investigation was made into solvents which could be used safely in conjunction with PPO foam. The results of this survey are summarized in Table 9-1.

Table 9-1. Effect of Various Solvents on PPO Foam

- | |
|--|
| <p>A. True solvents for PPO foam - Dissolve the foam more or less rapidly at room temperature</p> <p>chloroform
trichloroethane
methylene chloride
benzene
toluene
Cee-Bee C-105 (commercial polyurethane stripper)</p> <p>B. The following reagents partially dissolve PPO foam:</p> <p>methylethyl ketone
concentrated sulphuric acid</p> <p>C. The following reagents weaken, but do not visibly dissolve, PPO foam:</p> <p>hexane
vaseline
acetone</p> <p>D. The following reagents have no apparent effect on PPO foam:</p> <p>methyl alcohol
ethyl alcohol
water
aqueous acids
aqueous alkali
Uresolve-HF and -Plus (commercial polyurethane/epoxy strippers)
Forrest Products Laboratory etchant
Freon-TF</p> |
|--|

9.2.2.1 Polyurethane Methods. The urethane adhesives tend to be elastic but if bonded to properly etched aluminum are virtually impossible to peel. Their elasticity tends to cause them to resist high speed cutters and to fill abrasive discs.

A chemical method of repairing PPO foam panels which had been bonded to an aluminum substrate with a polyurethane adhesive, Crest 7343 was investigated. The objective was to strip off the damaged sections of PPO foam and adhesive without injury to the underlying aluminum substrate or to the adjacent areas of PPO foam and polyurethane adhesive. Based on previous knowledge of the susceptibility of PPO foam to attack by organic solvents, it was decided to limit this stripping investigation to alcoholic/aqueous reagents. Pieces of cured Crest 7343, Shore A hardness = 85^{+1} , were tested in a series of solvents and the hardness measured both wet and dry after 24 hours immersion, Table 9-2. The two commercial strippers, Uresolve-Plus and -HF, offer the greatest promise. These Uresolves are weakly basic, water soluble solutions, with flash points of 125°F and PH = 8 and 10.5, respectively. PPO foam is relatively unaffected by either of these Uresolves; only slight discoloration resulting from the 24-hour/73°F immersion. In addition, the Uresolves can be easily rinsed off the PPO foam. Unfortunately, the stripping action of the Uresolves was inadequate after 24 hours at room temperature. In order to enhance the stripping action, some time/temperature immersion tests were performed. The results are summarized in Table 9-3.

Aluminum panels to which 1.8 in. thick PPO foam sheets were bonded, were subjected to a combination of mechanical and chemical cleaning. Circular 12.7 cm (5 in.) in a diameter by 4.3 cm (1.7 in.) deep were cut into 4.6 cm (1.8 in.) thick PPO foam. Ninety-five percent of the PPO foam contained within this 12.7 cm (5 in.) diameter slit was removed mechanically. The bottom of the resulting 12.7 cm (5 in.) diameter cavity still contained a .25 cm (0.1 in.) of PPO foam. A 5.1 cm (2 in.) high metal liner was fitted snugly around the periphery of the cavity and sealed to the bottom with parafin wax. Uresolve-Plus was then poured into the cavity flushed with water and the metal liner pulled out. A second cavity was prepared in the same way and treated with Uresolve HF.

The results were relatively poor. Neither of the Uresolves have sufficient stripping action on Crest 7343 at room temperature to remove much of the 0.1 in. thick PPO foam/adhesive layer. In addition, the parafin seal along the bottom of the metal liner leaked. This leakage, however, resulted from the inability of the relatively high melting wax to penetrate into the rough, cold PPO/adhesive interlayer mounted on a cold aluminum substrate. Warming the substrate prior to pouring, or use of a lower melting range wax would solve this problem. Most aqueous etchants are compatible with PPO foam. For example, the sodium dichromate/sulphuric acid/water solution known as Forest Products Laboratory Etchant had no effect on PPO foam at either room or elevated (150°F) 339K (150°F) temperatures. This Forest Products Laboratory Etchant is also compatible with the waxes (parafin wax, green tape, etc.) which might be used to seal the metal liner to the PPO foam/adhesive base.

Table 9-2. Effect of Various Reagents on Polyurethane Adhesive

	Crest 7343 Hardness	
	Wet	Dry
Acetone	80	85
1% KOH in CH ₃ OH	65	84
1% KOH + 1% H ₂ O in CH ₃ OH	72	84
1% KOH + 2% H ₂ O in CH ₃ OH	65	84
1% KOH + 5% H ₂ O in CH ₃ OH	65	82
1% KOH + 10% H ₂ O in CH ₃ OH	67	81
1% KOH + 25% H ₂ O in CH ₃ OH	70	84
Uresolve-Plus	58	66

Table 9-3. Effect of Time/Temperature on Uresolve Stripping
Action on Crest 7343

Temperature	Time	Uresolve-Plus	Unresolve-HF
73°F	24 hr.	softened but not strippable	hard and tough; not strippable
73°F	48 hr.	will strip but not cleanly	strippable but not cleanly and with difficulty
100°F	1 hr.	unchanged	unchanged
100°F	3 hr.	softened	unchanged
100°F	5 hr.	soft; can be manually scraped off	hard, adherent.
100°F	7-1/2 hr.	soft; can be manually scraped off	hard, adherent.
100°F	13 hr.	can be partially wiped off	can be peeled off but a residue remained which cannot be removed
150°F	1-1/4 hr.	soft; can be scraped off	hard; can be partially scraped off
150°F	2 hr.	readily scraped off but no dissolved	hard; can be peeled off but a residue remains
150°F	4-3/4 hrs.	readily cleaned by wiping, but still not dissolved.	can be cleaned by wiping

Development of a chemical stripping method for repairing urethane bonded PPO foam panels was not completely successful. If the Uresolves are used as the stripping agents, elevated temperatures are definitely required. This will require the use of a "pumped" continuous flow system, at least for repairs on all vertical or overhead PPO foam bonded areas. However, due to safety considerations, the Uresolve temperature should be maintained below the 325K (125°F) flash point. Under these conditions, even with a pumped system, several hours might be required to chemically strip off the adhesive underlying the PPO foams.

A faster, more efficient stripping agent than the Uresolves might do a better job. However, the familiar commercial urethane stripping agents which are more effective than the Uresolves (such as Cee Bee #105 or Pennwalts "Wedge") also have a catastrophic effect on PPO foam. It is mandatory that any chemical stripper used be incapable of massive PPO foam degradation in case of an accidental spill. Finding a more effective polyurethane adhesive stripper than the Uresolve will require further research.

9.2.2.2 Epoxy Cleaning. Epoxy adhesives tend to be more brittle and harder than urethanes thus lend themselves to cutting and abrading. This approach was used effectively for Hysol 394.1. To accomplish this, 5.1 cm (2.0 in.) diameter abrasive disks were used. Figure 9-10 shows the two types of abrasive disks used along with the air motor and rubber backing disk. The abrasive disks snap into the rubber backing disk for quick and easy changing. The first disk used in removing the epoxy adhesives is a 60 grit sand paper which removes the bulk of the bond line. Then, a



Figure 9-10. Abrasive Disks and Air Motor for Cleaning the Bond Line

disk of scotch brite abrasive material is used to finish the removal of the epoxy adhesive. To contain the dust generated by the grinding, a clear plastic bag that fits around the air motor and tapes to the mylar sheet covering the foam is used. Figure 9-11 shows the dust containment bag in use. The bag is tapered with the small end clamped around the air motor and the large end taped to the mylar sheet. The operator can easily see through the bag and extra abrasive disks inside the bag can be easily snapped onto the rubber backing disk without opening the bag. After cleaning the bondline, the cylindrical blade is removed and the repair hole is ready to have a new piece of foam bonded in place. A finished repair hole is shown in Figure 9-12.

9.2.3 FOAM REPAIR INSERT. Bonding a new piece of PPO foam into the repair hole is accomplished by compressing an oversize foam plug, inserting the plug into the hole and then releasing the plug from compression. The tools for compressing and inserting the repair plug are shown in Figure 9-13 along with a foam repair plug. The compressor is expanded and then placed around the foam plug and tightened by means of ratchet. The disk with a straight handle is used to push the foam out of the compressor into the insert tool. Figure 9-14 shows the foam plug in the insert tool in the repair hole ready for extraction of the insert tool. Before inserting the foam plug, the aluminum surface at the bottom of the repair hole and the bottom face of the foam plug are coated with Hysol ADX 394-1 epoxy adhesive. A styte 104 glass scrim cloth is then applied to the foam plug and trimmed and the plug is ready for insertion. To hold the foam plug against the aluminum tank wall during bond cure, vacuum bagging technique was used. The adhesive back mylar sheet applied to the foam surface at the outset of the repair procedure provides a surface to which vacuum a bag is applied. Figure 9-15 shows the vacuum bag system over a repair area.

9.3 REPAIR JOINT TESTS

A thermal conductivity test specimen of the compression butt joint fabricated from one of the best performing configuration screening thermal conductivity test specimens. In this manner, the thermal performance of the joint can be compared directly with the performance of the same specimen without a joint. The specimen used was 72-41; 24.9 mm (0.98 in.) thick. A 127 mm (5.00 in.) hole was cut into the center of the specimen using a rotating cutter, Figure 9-16. The cutter was machined from aluminum and has 0.635 mm (0.025 in.) walls and a smooth, sharp cutting edge. A 25 mm (1.0 in.) arbor machined into the top of the cutter allows the cutter to be mounted vertically. The rotating cutter was used to cut the hole to within 1.27 mm (0.050 in.) of the aluminum foil on the heater. Then the foam in the hole was cut away by hand and the adhesive bond to the aluminum heater was peeled away leaving an extremely clean cut and heater surface.

An oversized diameter plug is cut from material of the same foam panel as the original thermal conductivity specimen (see Figure 9-17), allowing for residual edge

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Figure 9-11. Dust Containment Bag Attached to the Air Motor and Mylar Cover Sheet

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Figure 9-12. Finished Repair Hole

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Figure 9-13. Tools for Compressing and Inserting Repair Plugs and a Foam Plug

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Figure 9-14. Foam Plug Inserted in a Repair Hole

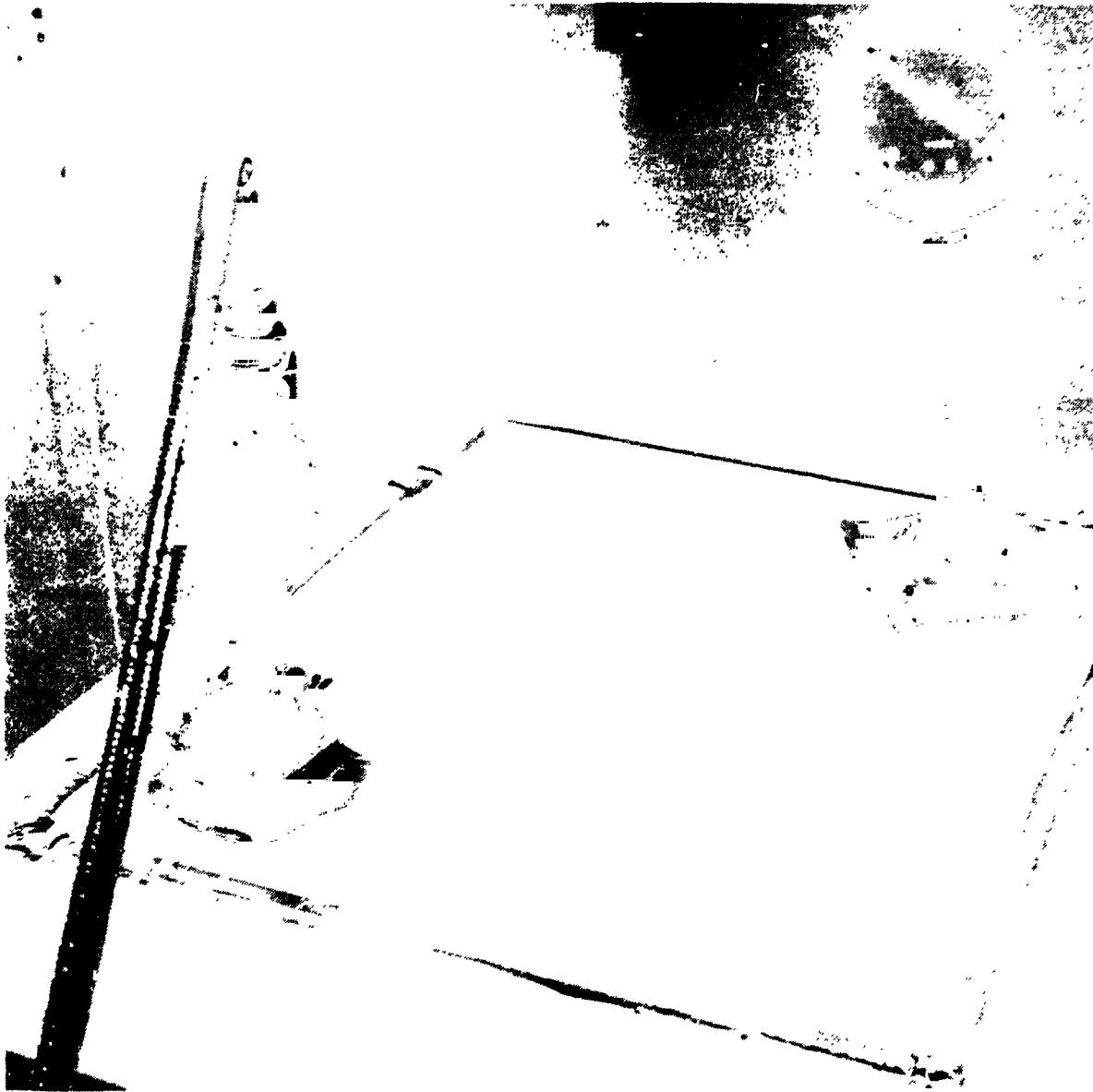


Figure 9-15. Vacuum Bagging System for a Panel Repair

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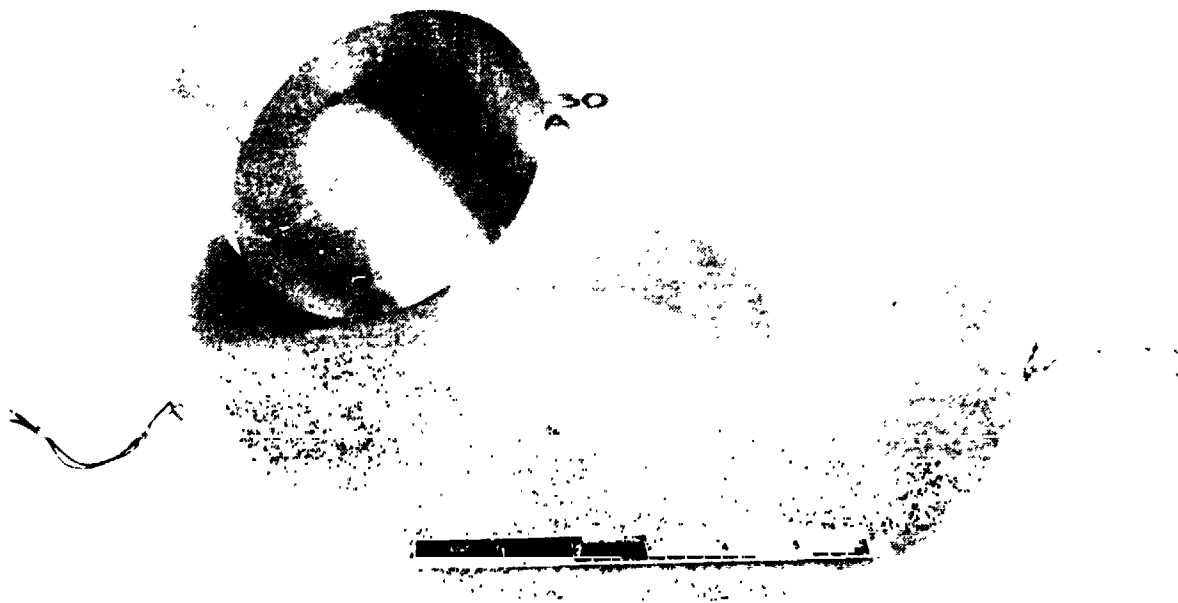


Figure 9-16. Panel Joint Specimen and Hole Cutter

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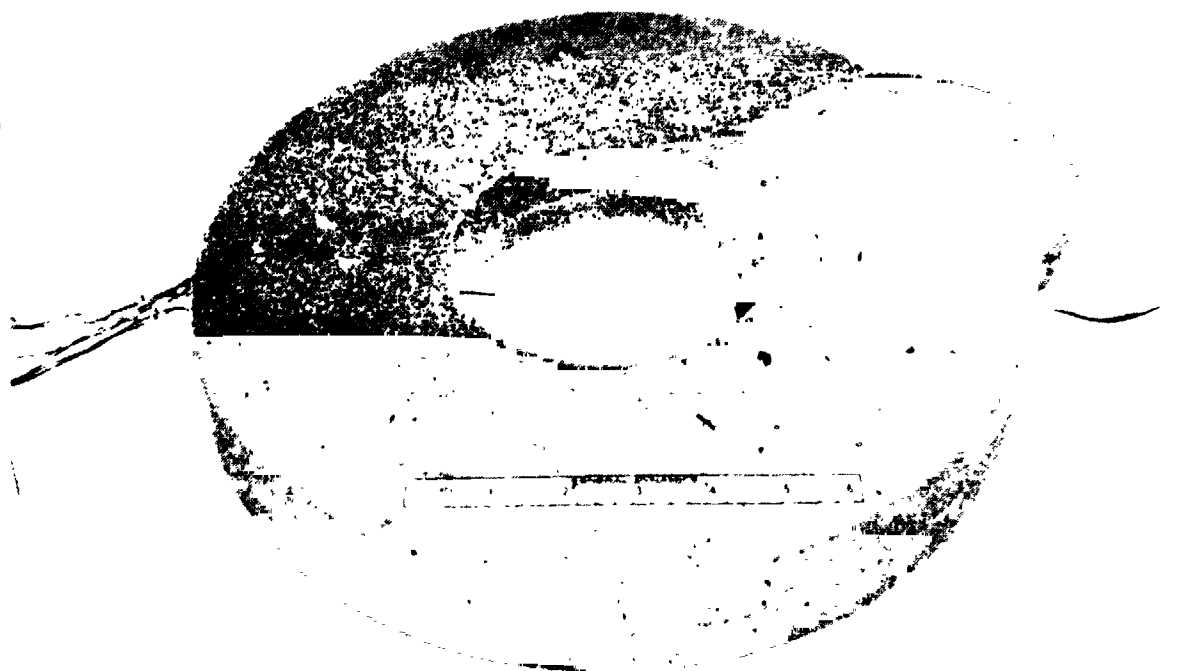


Figure 9-17. Panel Joint Specimen and Foam Plug

compression. The foam plug is compressed, using the tool shown in Figure 9-18. Adhesive is applied to the aluminum heater and the bottom of the foam plug and the plug inserted into the hole. The compression tool is extracted while holding the plug in place. Finally, the specimen is vacuum bagged and left to cure over night. Figure 9-18 is the finished joint specimen. Except for the ink outline of the hole, the joint is indistinguishable from the original, continuous piece of foam.

Thermal conductivity test results for the joint repair panel is shown in Figure 9-19. The 'previous calorimeter data' was that run on the original thermal conductivity specimen in 1972. The specimen was re-run as a baseline point in September 1973. There was a nominal 20 percent increase in apparent thermal conductivity between the original and baseline run. There was no discernible reason for this, i. e., no physical damage or non-uniform temperature readings. The 4% compression was run in November 1973 and the 8% compression in January 1974. There is an obvious improvement in performance from the 4% compression repair to the 8% compression repair. The greater the compression, the better joint from the standpoint of hydrogen entry and convection. The net degradation, however, is exceptionally good considering the specimen design and conditions to which it had been subjected. The specimen consists of two pieces of PPO foam bonded to a thin flexible heater. This non-rigid specimen had been bonded originally, then cut, cleaned and repaired twice; immersed in liquid hydrogen six different times and thermal cycled ten times. The net averaged degradation from the baseline to the 8% compression repair test was only 18 percent. Repair of PPO foam on a rigid base such as the shuttle expendable tank should therefore not represent a significant problem from the standpoint of performance degradation.

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Figure 9-18. Finished Panel Joint Specimen and Foam Plug Compressor

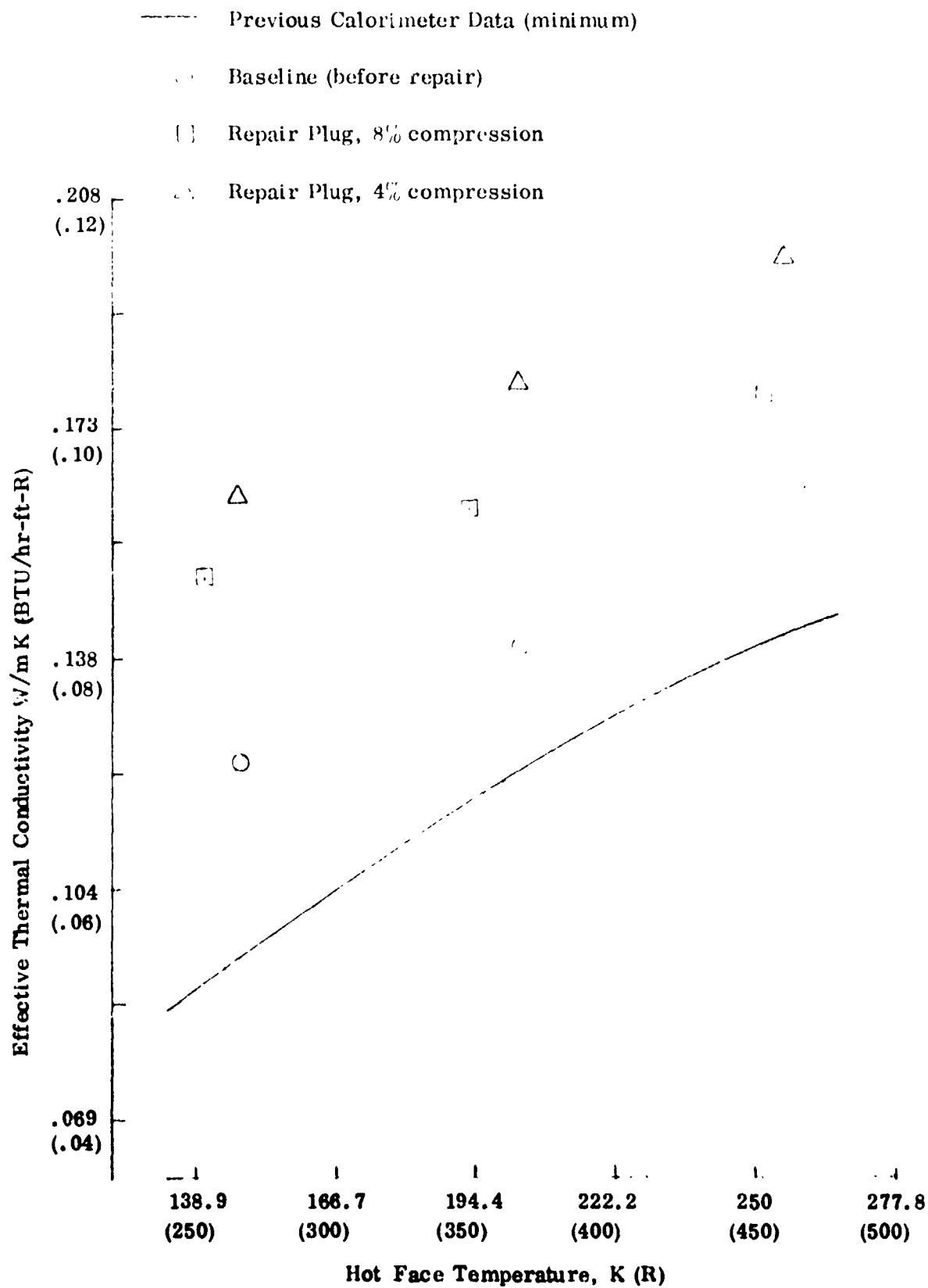


Figure 9-19. PPO Foam Repair Test Results

SECTION 10

ADHESIVES AND TANK SURFACE PREPARATION

Since PPO foam is an internal insulation system, the tank wall and adhesive bondline will not normally encounter temperatures below 200K (-100F) during a typical mission cycle. However, should a temperature depression occur at some point on the tank it is imperative that the adhesive system prevent the propagation of small, localized failures into a major system delamination. Consequently, the adhesive system must properly be compatible with the foam and tank wall over the full system operating temperature range, 21 to 450K (-423 to 350F). In the past polyurethane adhesives have been employed as the principal foam insulation bonding agent due to their high strength and ductility at cryogenic temperatures. However, the maximum operating temperature is usually limited to 366 to 394K (200 to 250F), above which the strength falls off rapidly. The addition of small amounts of coupling agents has improved the high temperature performance, but the polyurethanes remain marginal at best at the 450K (350F) temperature extreme. Also, the catalyst used with the polyurethane adhesive, 4,4-methylene-2-chloraniline, was termed carcinogenic by the Occupational Safety and Health Administration, and thus, its use would require extremely complex cleanroom procedures. Therefore the investigation described in this report was designed to gather data on a large number of adhesive systems, evaluate these data, select several promising candidates for comparison with a typical polyurethane adhesive, and finally to select a preferred system and perform an indepth evaluation of its performance with PPO foam over the 21 to 450K (-423 to 350F) temperature range.

10.1 LITERATURE SURVEY

The available current literature dealing with the use of adhesive systems on aerospace vehicles has been reviewed to gather all available data on the various systems and to assemble information on any new, high-potential adhesives which may not have been extensively evaluated. These data have been carefully reviewed and four adhesive systems have been selected for a screening program designed to evaluate their relative performance and to permit the selection of one system for detailed investigation with PPO foam.

10.1.1 ADHESIVE SYSTEM SURVEY. Prior to the selection of candidate adhesive systems for use with PPO foam over the temperature range 21 to 450K (-423 to 350F), an extensive search of the literature was conducted. Principle sources of data on adhesive systems for cryogenic application were investigations performed by a number of aerospace companies including McDonnell-Douglas, Martin Marietta,

North American (Rockwell International) as well as both the Ft. Worth and Convair Division of General Dynamics. Table 10-1 summarizes the results of this investigation. Available information on a total of fifty-four candidate systems is presented. Several trends which were found during an analysis of the data are itemized below.

1. Aeromatic and heterocyclic polymers, such as phenolic, PI, or PBI adhesives exhibited excellent strength characteristics throughout the temperature range of interest. However, all of these systems required elevated temperature cure.
2. Epoxies performed quite well at elevated temperatures but tended to be glass like, or brittle, at liquid hydrogen temperature.
3. Polyurethane exhibited outstanding strength at cryogenic temperatures but were marginal or unacceptable at 450K (350F).
4. Polysiloxanes have been used extensively over the temperature range of interest, but in general exhibited much lower strengths than did the other types of polymers.

The two primary ground rules used in selecting adhesive systems for preliminary screening tests were the following:

1. The systems must give an indication of providing adequate performance over the 21 to 450K (-423 to 350F) temperature range.
2. Adhesives curing at room temperature are preferred. However, an oven post cure of 333K (140F), simulating shop conditions for the cure of extremely large structures, would be allowed.

10.1.2 SELECTED CANDIDATE SYSTEMS. Based on these ground rules and an analysis of the information gathered on the fifty-four candidate systems, the following four systems were selected for the screening evaluation:

1. Crest 7343/Z6040. This polyurethane adhesive system is well known for its outstanding properties at cryogenic temperatures. The addition of the Silane coupling agent has greatly improved the properties at elevated temperatures. Although its performance with PPO foam is still marginal at 450K (350F), the adhesive does appear to possess adequate strength at 422K (300F). This system has been included in the screening evaluation to (1) serve as a baseline against which the remaining systems can be compared, and (2) evaluate the effect on its performance of the addition of a glass scrim.

Table 10-1. Results of Literature Survey

ADHESIVE	CURE TEMP.	CONVAIR	NAR	MDAC	MARTIN	GENERAL COMMENTS
Leikoweld 109 (Leifflingwell Chem.)	R. T.	Passed thermal shock tests to -423°F, adequate to 300°F	Leading candidate, adequate to +300°F, OK cryogenic	-	Good system but not chosen due to cryogenic properties (RTV560 was chosen in this study)	Basically satisfactory but a marginal material due to poor 300-350°F performance not selected (N.S.)
Leikoweld 211A/62 (Leifflingwell Chem.)	300°F	-	-	Good performance, selected as best in this study	Good material, fairly level strength from -423°F to 350°F	Excellent performance, but 300°F cure requirements rule out this system. Not selected (N.S.)
934 (Hysol)	R. T.	Good properties to 350°F, marginal -423°F, has some times failed thermal shock/cycling at -423°F. Good candidate	Good performance at 300-350°F, may be marginal at -423°F. Good candidate	-	-	Candidate, base material. Good reference point will be included
7343/ Z6040 (Crest Products)	R. T.	Best candidate to date, excellent -423°F, marginal at 350°F	Promising material, excellent at -423°F, marginal at 350°F. One of the best materials when silane modified	Used without Z6040, therefore low 300°F performance	-	Promising candidate. Will be included. Much data available from work at Convair Aerospace
HT-424 (American Cyanamid)	300-350°F	Good cryogenic, excellent 350°F, passes thermal shock, high T cure is biggest drawback	-	Good performance over full range of temperature	Good results over entire temperature range	Generally OK with less potential than PJ's, N.S. since it is of little interest to NASA and requires a 350°F cure
Melbond 329 (Whittaker)	350°F	Used successfully at -423°F although somewhat glossy. Good at +350°F. 350°F cure is required	-	-	-	Excellent performance, but high T cure is the reason for rejection
FM-34 (American Cyanamid)	350°F	Good -423°F to +350°F with this cure. Passes thermal shock/cycling. At least 350°F cure needed	-	Good strength at all temperatures	-	Inferior to 934, so N.S.
951 (Hysol)	350°F	Same as above, but more reliable in cure. Readily processed in vacuum bag	-	-	-	Performance is outstanding, unfortunately 350°F cure rules out this system. N.S.
BR-34 (American Cyanamid)	350°F	Solvent may attack foam. Otherwise same as FM-34	-	-	Rigidity at low temp. Not chosen, high temp. cure required	Inferior to 934, so N.S.
BR 90 (Am. Cyanamid)	-	-	-	No outstanding qualities	-	N.S.
5526 (Eponylite)	350°F	-	-	Good performance selected in this study, but more wt. needed for strength equal to Z11A, hard to apply	-	Lower strength than others, elevated temperature cure required. N.S.
Epibond 8510 (Furane Plastics)	-	-	-	Not as good as many others	Glassy at low T., not outstanding	N.S.
X-87285 (Furane Plastics)	-	-	-	Good performance, but lower strength than 5526 & Z11A	-	Not selected due to low strengths at 300°F

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Table 10-1. Continued

X-87284 (Furane Plastic)	R. T.	-	-	Not up to 37285 performance	-	Low strength, N S
4822 (Epoxyite)	R. T.	-	-	Performance less than that of 5526	-	N S, inferior
810 (Epoxyite)	R. T.	-	-	Performance not up to 5526	-	Not as good as 5526, N S
Eccobond 276 (Emerson & Cuming)	-	-	-	Not outstanding	-	N S.
Eccobond 104 (Emerson & Cuming)	-	-	-	No special merit	-	N S.
Glon On (Thermo Resist)	150°F	-	-	Average product, weak at cryogenic T.	-	N S.
828/CL (Hynell)	R. T.	-	-	Not especially promising	-	N S.
Lefkowitz 108 (Lefkowitz Co.)	350°F	-	-	Not up to 109	-	Other Lefkowitz products are superior, N S.
Lefkowitz 2232-27/22 (Lefkowitz Co.)	-	-	-	Not up to 211A	-	Other Lefkowitz products are superior, N S
5527 (Epoxyite)	-	-	-	Not as good as 5526	-	N S.
A-60 (Armstrong)	-	-	-	Generally poor performance	-	N S.
Thermadite 17 (Whitaker)	350°F	-	-	Not as good as BR34-FM34 products	-	Inferior to 951, so N S
3170/7123 (Cresco)	R. T.	-	-	Performance below that of 7343	-	Not outstanding, glassy at low T
3558 (3M)	-	-	-	-	-	Failed A1 dome cycling test
H-21 (Epoxy Tech.)	-	-	-	-	-	Typical epoxy, not out- standing, glassy at low T.
H-61 (Epoxy Tech.)	-	-	-	-	-	Typical epoxy, not out- standing, glassy at low T
H-76 (Epoxy Tech.)	-	-	-	-	-	Typical epoxy, not out- standing, glassy at low T.
H-75 (Epoxy Tech.)	-	-	-	-	-	Typical epoxy, not out- standing, glassy at low T
RTV 540 (G.E.)	R. T.	-	-	-	-	Best system in this study, excellent -423°F, adequate at 300°F

Work at Ft. Worth shows
adequate properties from
-423 to +350°F with about
150 psi lap shear at 350°F.
An excellent candidate.

Table 10-1. Continued

WD 5332 (Am. Cyanamid)			Typical epoxy, not outstanding	N. S.
7344/7119 (Lafayette Co.)			Glassy at low T, not outstanding	N. S.
RTV 156 (G. E.)	R. T.		One of the selected system, excellent -423°F, marginal at 300°F, excellent low T peel	Not selected due to unreasonably short pot life unless work is done in N ₂ atmosphere. This is impractical for our purposes
RTV 583 (Dow Corning)	R. T.		Failed Al dome cycling	N. S.
RTV 589 (Dow Corning)	R. T.		Failed Al dome cycling	N. S.
RTV-96-052 (Dow Corning)	R. T.		Extremely low R. T. strength	N. S. due to low R. T. strength
Uralite 312 (Resolene)				N. S. as it is not outstanding
Imidite 850 (Whittaker)	to 600°F	Excellent strength	Fairly good performance at all temperatures	Cure problems, volatiles, expensive temperature rules out this resin
AF 31 (3M)	350°F		Good performance at all temperatures	Not up to PI performance, so with high T cures required, N. S.
AF 300 (3M)			Good strength over temp. range, glassy at low T, elevated cure required	Glassy at low T, not up to PI performance, elevated temperature cure required, N. S.
EC-2:16 (3M)			Poor at both 300°F and -423°F	N. S.
AF 41 (3M)	350°F		Good strength over temp. range, glassy at low T, elevated cure required	N. S.
AF 126-2 (3M)	225 - 300°F		same as above	N. S.
Methylbond 400 (Whittaker)	350°F		Good strength cryogenic, but may be glassy	N. S.
FM-96 (Am. Cyanamid)			Glassy at -423°F, low strength at cryogenic T	N. S.
FM-1000 (Am. Cyanamid)			Fairly good performance throughout temperature range	N. S.

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Table 10-1. Continued

FM 123-2 (Am. Cyanamid)	-	-	Not outstanding, loses strength rapidly at 300°F. glassy at low T.	-	N.S.
BR 96 (Am. Cyanamid)	-	-	Same as FM 96	-	N.S.
BR 92 (Am. Cyanamid)	-	-	Poor at both ends of temperature range	-	N.S.
93-046 (Dow Corning)	R. T.	Notes promising as RTV-560 in Ft. Worth work Lower strengths at elevated temperatures	-	-	N.S., inferior to RTV-560
Martin SLA-561 (Martin Marietta)	R. T.	Inferior to 93-046 and RTV-560	-	-	N.S., inferior to RTV-560
3515 (3M)	R. T.	Performance adequate but inferior to 7343/26040 and 934, especially at elevated temperatures	-	-	N.S.
Hysol 394 (Hysol)	R. T.	New candidate; vendor data is all that is available	-	-	Candidate system, a flexibilized or toughened version of 934 which should be superior at -423°F

2. Dow Corning RTV-560. This polysiloxane system has been used successfully over the temperature range of interest by a number of firms (Table 10-1). By comparison its strength is lower than that of the polyurethanes at cryogenic temperatures and lower than that of the epoxies at elevated temperatures. Nevertheless its performance appears to be adequate for use with PPO foam.
3. Hysol EA934. This epoxy adhesive has become a standard of the aerospace industry. The system handles easily and possesses excellent characteristics at elevated temperatures. Like other epoxies, however, it exhibits a tendency toward brittleness at liquid hydrogen temperature. Additional data are required at cryogenic temperatures to make a valid comparison of its properties with those of the other candidate systems.
4. Hysol ADX 394-1. The manufacturer claims that this new epoxy adhesive is an improved version of the widely-used EA934 system. It is said to possess elevated temperature performance equivalent to that of EA 934 with improved toughness or flexibility at cryogenic temperatures.

10.2 CANDIDATE SCREENING EVALUATION

The selected adhesive systems have been screened to determine their relative performance with PPO foam insulation. Based on these preliminary tests one system has been selected for a detailed investigation with the foam over the 21K to 450K (-423 to 350F) temperature range.

10.2.1 TEST PLAN AND CONDITIONS. The screening test program was designed to determine the basic material strength of the various candidates and to evaluate their performance with PPO foam under the most critical loading conditions. A list of the screening test appears in Table 10-2.

Tensile lap shear tests were performed on each system at 20, 294, and 450K (-423, 70, and 350F) using ASTM Standard D1002-64. The 2024-T3 aluminum adherends were prepared by solvent wiping followed by light abrasion and a Pasa Jell 105 etch. The adhesive was applied to both surfaces and 76 μ m(0.003-in) wires were placed in the bondline to control its thickness. The specimens were cured for 24 hours at room temperature under a 14 kN/m² (2 psi) load followed by a four-hour 333K (140F) post cure. The Crest system with the glass scrim was prepared in a similar fashion. The scrim was not preimpregnated but was simply sandwiched between the adhesive-coated adherends. The foam specimens were bonded in a similar manner. PPO foam panels were sliced into specimens using a horizontal band saw. A light coat of adhesive was applied to the face of the foam and allowed to stand until tacky before bonding to minimize adhesive penetration into the foam.

Table 10-2. Candidate Adhesive Screening Program

	Temperature - K (F)			Specimen Size, cm (in.)	Remarks
	21 (-423)	78 (-320)	294 (70)	450 (350)	
1. Lap Shear	X		X	X	Test 3 specimens at each temperature, 3 x 3 = 9 tests.
2. Climbing Drum Peel		X	X		Test 3 specimens at each temperature, 3 x 2 = 6 tests.
3. Load Deflection		X			Bond 4 specimens in 8.9 x 16.5 x .318 cm (3.5 x 6.5 x .125) T 81 aluminum plate, deflect 2 at 78K (-320F), 50 deflections, 1.13 kN (254 lb) load. See Below.
4. Thermal Cycle		X	X	X	Thermal cycle all 4 specimens at no load. 1. 294K (70F) to 78K (-320F), 6 cycles 2. 294K (70F) to 450K (350F), 6 cycles 3. 294K (70F) to 78K (-320F), 2 cycles
5. Load Deflection		X		X	Test all 4 specimens at both temperatures 50 deflections, 1.13 kN (254 lb) load.

Tensile lap shear test results for the various candidates are listed in Table 10-3 and are plotted in Figure 10-1. The addition of the scrim to the Crest polyurethane system had no appreciable effect on the ultimate lap shear strength. Performance at 450K (350R) is still marginal at best. As expected the RTV system displayed low strengths although the value at 450K (350R) appears to be adequate for a PPO foam application. The two epoxies exhibited similar performance at the temperature extremes with the 394 system approximately 20 percent higher at room temperature. The seemingly low strength values at 21K (-423F) are more than adequate for use with PPO foam.

Two other types of screening tests were conducted which include climbing drum peel and "load deflection." Peel is run to determine the torque required to separate the bonded system when subjected to a peeling load. Load deflection evaluates the ability of the system to withstand transverse loading at various temperatures before and after being thermally cycled and shocked. Peel test specimens were bonded as discussed above and tested per ASTM D1781-62. The test apparatus and a failed specimen are shown in Figure 10-2.

10.2.2 TEST RESULTS. The Crest samples indicated the highest peel strength,

and they were also the only specimens where the failure occurred clearly in the PPO foam. All of the foam specimens were cut from panels having a nominal density of 40 kg/m^3 (2.5 lb/in^3). The specimens with the glass scrim indicated nearly a 50 percent improvement in peel strength at ambient temperature as compared to the value at 78K (-320F), whereas the Crest specimens with no scrim exhibited a horizontal peel strength curve between the same two temperatures. Both of the epoxies displayed peel strength that increase with temperature. The adhesive rather than the foam was the system

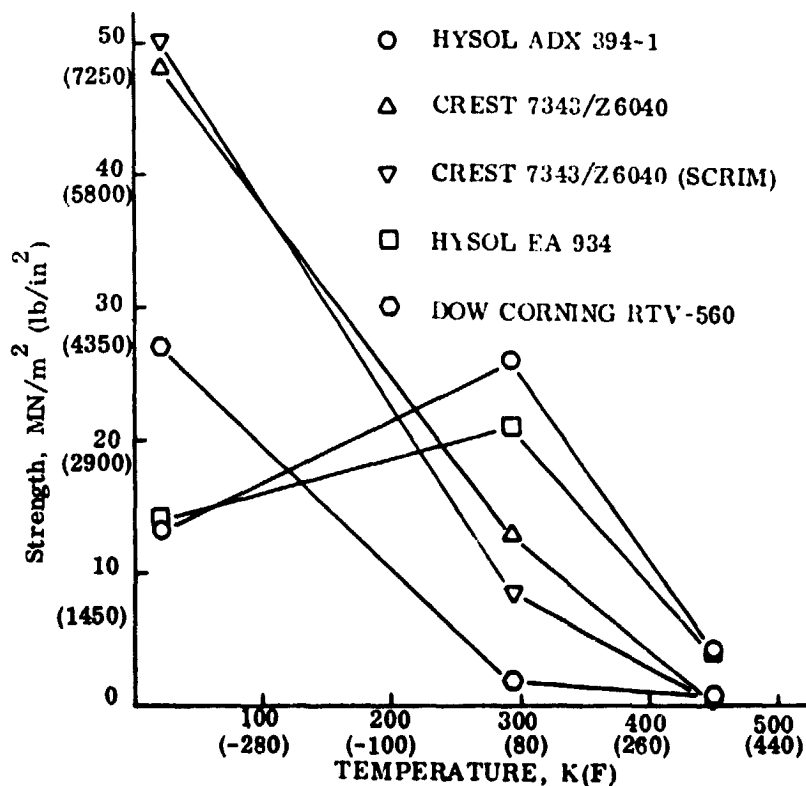


Figure 10-1. Candidate Systems Lap Shear

Table 10-3. Results of Adhesive Screening Evaluation

TEST	TEMP K (F)	DOW CORNING RTV 560	CREST 7343/Z-6040	CREST 7343/Z-6040 104 GLASS SCRIM	HYSOL EA 934	HYSOL ADX 394-1
1 Lap Shear	21 (-423)	27,000 (3,920) 1,940 (281) 841 (122)	* 48,000 (7,000) 12,800 (1,850) 14 (2)	* 50,000 (7,300) 8,410 (1,220) 31 (4.5)	14,000 (2,030) 21,000 (3,040) 3,860 (560)	13,400 (1,950) 25,700 (3,730) 4,100 (595)
	294 (70) 450 (350)					
2 Climbing Drum Peel	78 (-320)	80 (18) 23 (5.2)	98 (22) 102 (23)	93 (21) 133 (30)	24 (5.3) 53 (12)	33 (7.5) 62 (14)
	294 (70) 450 (350)					
Load Deflection	78 (-320) 294 (70) 450 (350)	One Failure At 450K After Thermal Cycling	Two Failures At 450K After Thermal Cycling	One Failure At 450K After Thermal Cycling	One Failure At 78K After Thermal Cycling	No Failures

* Adherend failure

1 kN/m² (psi)

2 N · m/m (lb · in/in) of width

which failed in all of the epoxy peel tests. The slope of the RTV 560 curve was negative as the maximum strength dropped some 70 percent as the temperature was increased from 78 to 294K (-320 to 70F). The mode of failure of the RTV system at 78K (-320F) was unusual. As load was applied the adhesive elongated until it suddenly "popped" off of the foam with the surface profile of the foam clearly mirrored on the adhesive surface.

Load deflection tests are performed on the specimen configuration shown in Figure 10-3. A $7.6 \times 12.7 \times 4.6$ cm ($3 \times 5 \times 1.8$ in) block of PPO foam is bonded to an $8.9 \times 16.5 \times 0.3$ cm ($3.5 \times 6.5 \times 0.125$ in) 2219 aluminum plate using the procedures described previously. Two load deflection specimens are pictured in Figure 10-4. Four specimens were prepared for each of the candidate adhesives. Two were then deflected 50 times at 78K (-320F), all four were thermally cycled as described in

Table 10-2, and finally all four were deflected 50 times at both 78 and 450K (-320 and 350F). A transverse load of 1.13 kN (254 lb) was applied at the midpoint of the specimen at the rate of four cycles a minute resulting in a deflection of 3.8 mm (0.15 in). This deflection was determined to produce yield strain in the aluminum, the maximum bondline strain to which the adhesive would ever be subjected. The specimens were inspected visually while being deflected and ultrasonically after bonding and after each series of deflections.

The results of the load deflection test are shown in Table 10-3. All of the specimens subjected to deflections at 78K (-320F) before thermal

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Figure 10-2. Peel Test Apparatus and Specimen



Figure 10-3. Load Deflection Test Specimen Schematic

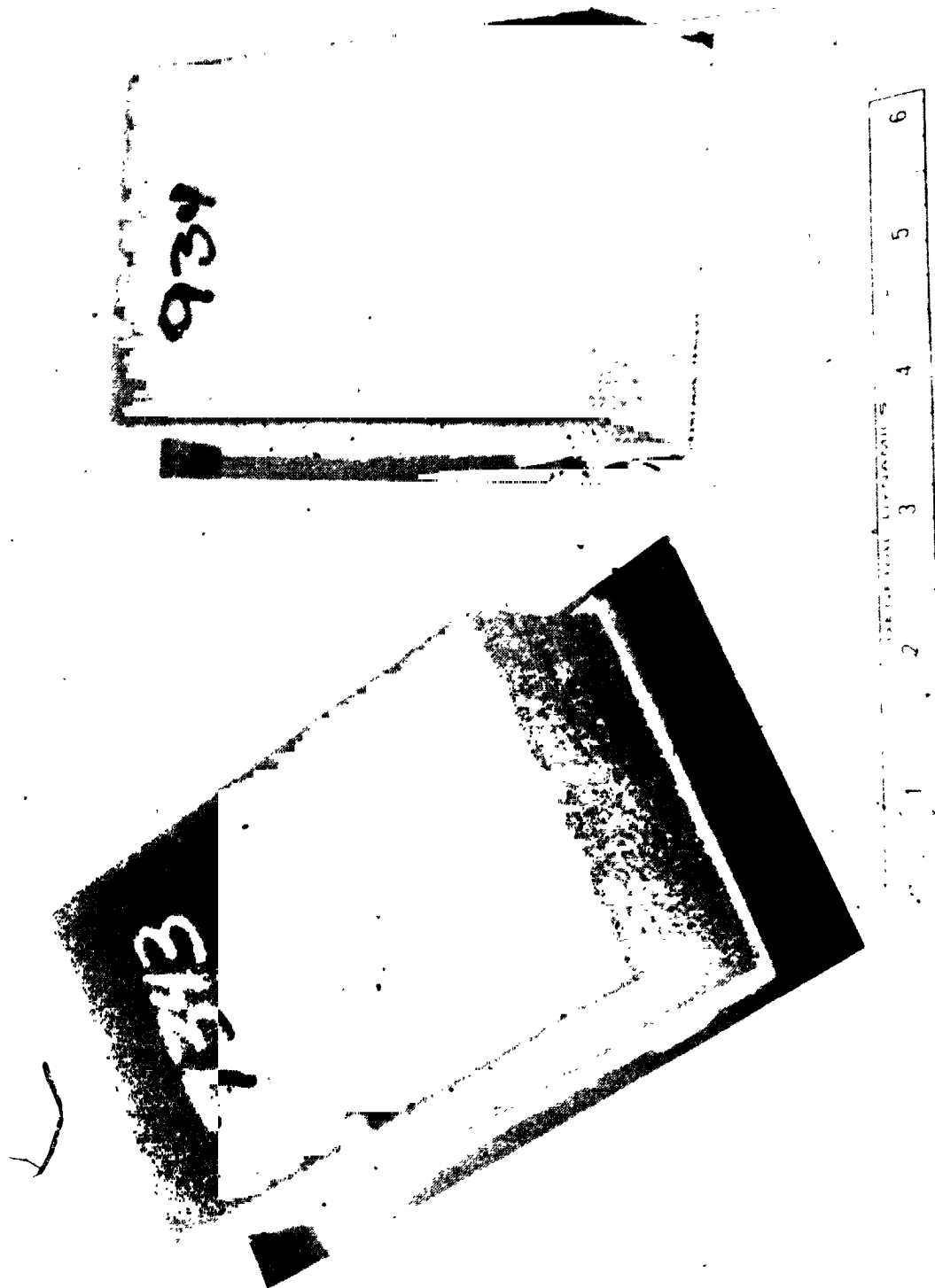


Figure 10-4. Load Deflection Specimens

cycling survived with no detectable delaminations. Subsequent to the cycling at least one specimen from each of the Crest and Dow Corning groups delaminated during the 450K (350F) deflection cycle. One Hysol EA934 specimen failed during the deflection cycle at 78K (-320F). However, none of the four Hysol ADX 394.1 specimens suffered delamination during the 78 and 450K (-302 and 350F) testing. Success at the higher temperature was expected, and success at the lower temperature tended to support the manufacturers claim that the 394-1 epoxy adhesive possesses improved ductility at cryogenic temperatures.

10.2.3 SELECTED ADHESIVE SYSTEM. Based upon its performance during the screening evaluation, the Hysol ADX 394-1 epoxy adhesive was selected for further indepth investigation with PPO foam. Due to the reduced ductility of epoxy adhesives at cryogenic temperatures, the decision was made to test the adhesives with the addition of a 104 glass scrim to give it every chance of meeting the system requirements. Since the scrim is not preimpregnated it adds only two additional steps to the bonding procedure; layup on one of the adhesive-coated adherends and trimming after curing. Another feature offered by the presence of the scrim is positive bondline thickness control, achieved by reducing the tendency of the adhesive to flow laterally due to the imposition of excessive or non-uniform pressure.

10.3 HYSOL ADX 394-1 INVESTIGATION

The Hysol ADX 394-1 adhesive with 104 glass scrim was selected for detailed investigation with PPO foam. A series of tests was performed on specimens before and after cycling 50 times over the temperature range of 21 to 450K (-423 to 350F).

10.3.1 TEST PLAN AND CONDITIONS. Table 10-4 outlines the test program for structurally evaluating the PPO foam insulation Hysol ADX 394-1 adhesive system. Two identical sets of specimens were prepared. The first set, which was not thermally cycled, was tested to serve as a baseline for comparison with data from the second set which had been subjected to 50 cycles between 21 and 450K (-423 and 350F) before testing. In addition to tensile lap shear and climbing drum peel tests described in Section 10.2, PPO foam core shear, face tension, and cyclic monostrain (dogbone) tests were performed at each of the four temperatures. The face tension and core shear tests were standard ASTM procedures (C297-61 and C273-61, respectively) designed to determine the load at failure of the foam/adhesive system under pure tensile and shear loading.

The cyclic monostrain (i.e., uniaxial) testing was designed to evaluate the structural integrity of the structure/insulation system combination under representative mechanical and thermal loading and environmental conditions in alignment with vehicle life cycle criteria. The design critiera and conditions of the Space Shuttle vehicle, including the influence of biaxial strain and compressive loading, were employed in determining the design and test criteria for the monostrain test specimens. The specimens were fabricated from 0.318 cm (0.125 in) 2219-T81 aluminum alloy plate which was cut

Table 10-4. Hysol ADX 394-1 Investigation Test Plan

- I. Use specified procedure to bond and cure specimens. Apply approximately 127 μ m (5 mils) adhesive to each surface, PPO foam and aluminum. Put scrim cloth in all bond lines.
- II. Run strength tests on first series of specimens as soon as bonded.

Test	20 (-423)	294 (70)	422 (300)	450 (350)	Specimen Sizes - cm (in)
1. Adhesive/Al, Lap Shear	X	X	X	X	2.54 x 2.54 x 0.254 mm (1 x 1 x 0.010)
2. PPO/Adh./Al, Face Tension	X	X	X	X	5.08 x 5.08 x 4.57 (2 x 2 x 1.8)
3. PPO/Adh./Al, Core Shear	X	X	X	X	5.08 x 15.25 x 2.03 (2 x 6 x 0.8)
4. PPO/Adh./Al, Peel	Xa	X	X	X	7.62 x 25.4 x 2.03 (3 x 10 x 0.8)
5. PPO/Adh./Al, Dogbone (400 cycles)	X	X	X	X	7.62 x 30.5 x 4.57 (3 x 12 x 1.8)

- III. Subject second series of specimens to 50 thermal endurance cycles, 20 to 450K (-423 to 350F).
- IV. Run strength tests on second series of specimens.

1. Adhesive/Al, Lap Shear	X	X	X	X	
2. PPO/Adh./Al, Face Tension	X	X	X	X	
3. PPO/Adh./Al, Core Shear	X	X	X	X	(Same)
4. PPO/Adh./Al, Cl. Drum Peel	Xa	X	X	X	
5. PPO/Adh./Al, Dogbone (400 cycles)	X	X	X	X	

Test Conditions

Run 20K (-423F) tests in LH₂.

Run 294K (70F) tests in air.

Run 422K and 450K (300F and 350F) tests in GN₂ (except Lap Shear, run in air). Test three specimens at each condition (each of three dogbones at each temperature).

Notes

- a. Climbing drum peel, run cryogenic tests at 78K (-320F) in LN₂.

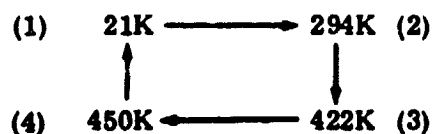
cut into rectangular pieces, 47.5×11.4 cm (18.7×4.5 in), with machined pin holes at each end. The PPO foam blocks were then bonded on and cured. Finally, the center section was notched down to the dogbone configuration, 5.08 cm (2.0 in) wide. A total of six monostain specimens were prepared and ultrasonically inspected. The test conditions and sequence are illustrated in Table 10-5.

10.3.2 THERMAL CYCLING. To be an acceptable component of the PPO foam insulation system, the adhesive must be capable of performing reliably in the face of wide environmental temperature variations. The criteria for the original Space Shuttle configuration was repeatable performance for up to 100 mission cycles where the bondline temperature could vary from 21 to 450K (-423 to 350F). Cycling from 21 to 450 to 21K (-423 to 350 to -423F) requires an elaborate test facility due to the requirement that the foam be isolated from air at 450K (350F). A compromise test program was selected. The test goal of demonstrating the effect of thermal cycling on bondline integrity was accomplished by subjecting a complete set of specimens described in Table 10-4 first to 50 cycles between 294 and 450K (70 and 350F) in a gaseous nitrogen environment followed by 50 additional cycles between 294 and 21K (70 and -423F) in a hydrogen environment.

10.3.2.1 Elevated Temperature. Four aluminum cases were fabricated to contain the test specimens for elevated temperature cycling. Each case contained a purge gas inlet and a thermocouple passthrough. Flexible purge lines were connected to the cases and to a GN₂ bottle. After thoroughly purging the specimens, the cases were sealed and an additional line was teed into the purge line and routed to a water flask to maintain a constant pressure in the system equivalent to a 5.1 cm (2 in) head of water during cycling. At any one time two of the cases were being heated in

Table 10-5. Cyclic Monostrain Test Conditions

Temperature K(F)	Load kN (lb)	Equivalent Strain m/m
21 (-423)	45.8 (10,300)	0.0034
294 (70)	44.7 (10,050)	0.0040
422 (300)	31.1 (7,000)	0.0028
450 (350)	29.8 (6,700)	0.0028



a circulating air oven while the other two were allowed to cool. The temperature in the oven was maintained at a level no higher than 456K (360F). While this resulted in a rather long heating cycle, it guaranteed limited local temperature overshoot at this critical cycle extreme. Upon reaching the specified temperatures the cases were switched and the cycle was completed. Using this procedure a total of 50 cycles between 294 and 450K (70 and 350F) were applied at an average time of 6.5 hours per cycle. At the completion of cycling the specimens were visually

inspected for discoloration and delamination. While the specimens had darkened somewhat, there was no indication of foam embrittlement or any other form of degradation. No delaminations were observed.

10.3.2.2 Cryogenic Temperature. At the completion of the elevated temperature cycling the specimens were transported to the Liquid Hydrogen Test Center, Site "B" for cryogenic cycling. The specimens were placed in baskets and mounted in a small vacuum jacketed test tank. Thermocouples were installed to monitor bondline temperatures. The tank was purged thoroughly with helium and evacuated to 69 kN/m² (10 psia) a total of five times prior to purging with gaseous hydrogen. During cycling care was taken to avoid thermally shocking the specimens since the bondline of a PPO foam system installed in a large tank would not normally be chilled at a high rate. The specimens were warmed by an ambient temperature gaseous hydrogen purge. Using this technique a cycle time of approximately four hours was achieved. The specimens were again visually inspected at the completion of cycling and no deterioration or delaminations were observed.

10.3.3 TEST RESULTS. Following the cryogenic cycling the second set of test specimens was subjected to the same test program (Table 10-4) as was the first set. Results of the various "static" tests for both the uncycled ("U") and cycled ("C") specimens are shown in Table 10-6. Lap shear strengths of the specimens are illustrated graphically in Figure 10-5. The strength of the cycled specimens is greater than that of the uncycled specimens at each test temperature. The elevated temperature exposure given the cycled specimens apparently caused a post curing which results in improved shear values over the whole temperature range. Figures 10-6 and 10-7 show the cycled specimens after shear testing at 21 and 450K (-423 and 350F), respectively. Compared with the results of the screening tests of 394-1 without the scrim, the cryogenic lap shear strength is considerably improved by the scrim. Results at the other temperatures are similar.

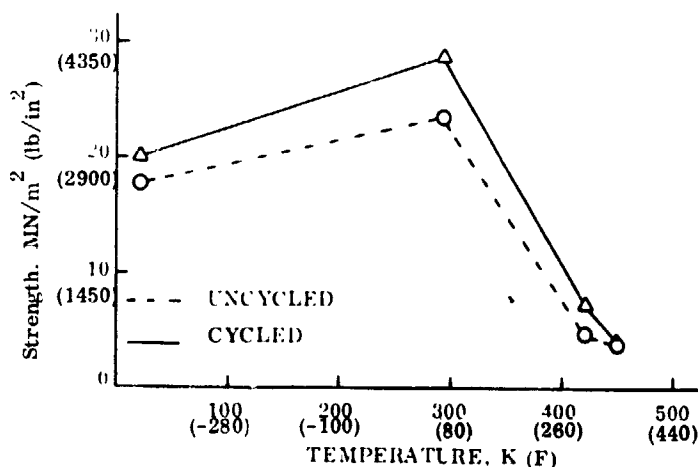


Figure 10-5. Lap Shear Strength as a Hysol AD394.1 Adhesive

Results of the foam face tension and core shear tests are illustrated in Figures 10-8 and 10-9. All of the specimen failures can be attributed to the foam component. At ambient and cryogenic temperatures the strengths of the cycled specimens averaged approximately 90 percent of those of the uncycled specimens. At 422 and 450K (300 and 350F) the reverse is true. Thus there

Table 10-6. Results of Hysol ADX 394-1 Investigation

	TEMPERATURE, K (F)							
	21 (-423)	294 (70)	424 (300)	450 (350)				
Lap Shear ²	U ¹ 18,500 (2680) 17,300 (2510) 17,900 (2600) Avg.	C 19,500 (2830) 23,000 (3340) 23,900 (3470) 23,200 (3370) 23,400 (3400)	U 23,000 (3340) 26,400 (3830) 29,300 (4250) 28,700 (4160)	C 30,300 (4400) 26,400 (3830) 29,300 (4250) 28,700 (4160)	U 4580 (664) 4840 (702) 4960 (720) 4790 (695)	C 7580 (1100) 7250 (1052) 7310 (1060) 7380 (1070)	U 3720 (539) 3650 (529) 3830 (555) 3730 (541)	C 4000 (590) 3860 (560) 3900 (566) 3920 (569)
Face Tension ²	- 855 (124) 896 (130) 876 (127) Avg.	1,076 (156) 440 (64) 765 (111) 765 (111)	1,670 (242) 1,690 (245) 1,700 (246) 1,680 (244)	1,590 (230) 1,610 (234) 1,460 (212) 1,550 (225)	670 (97) 896 (130) 862 (125) 807 (117)	896 (130) 910 (132) 883 (128) 896 (130)	630 (92) 650 (94) 590 (86) 630 (91)	570 (83) 580 (84) 610 (89) 590 (85)
Core Shear ²	440 (64) 400 (58) 450 (65) 430 (62) Avg.	460 (67) 410 (60) 340 (49) 410 (59)	360 (52) 370 (53) 350 (51) 360 (52)	310 (45) 320 (47) 330 (48) 320 (47)	160 (23) 170 (25) 170 (24) 170 (24)	210 (30) 190 (27) 190 (28) 190 (28)	- 130 (19) 130 (19) 130 (19)	140 (20) 150 (21) 170 (24) 150 (22)
Climbing Drum Peel ^{3,4}	* 38 (8.6) * 58 (13) * 55 (7.9) 43 (9.7) Avg.	* 39 (8.7) * 37 (8.4) * 40 (8.9) 39 (8.7)	* 62 (13.9) * 64 (14.3) * 61 (13.6) 62 (13.9)	* 48 (10.8) * 54 (12.1) * 57 (12.9) 53 (11.9)	25 (5.7) 61 (13.8) 83 (18.5) 56 (12.7)	43 (9.6) 48 (10.8) 48 (10.9) 46 (10.4)	22 (5.0) 16 (3.7) 24 (5.4) 21 (4.7)	12 (2.7) 13 (2.9) 15 (3.4) 13 (3.0)

1 U - uncycled, C - cycled

2 kN/m² (psi)

3 78K (-320F) rather than 21K (-423F)

* Adhesive failure; foam failure in all other (except lap shear)

4 N·m/m (lb·in/in) of width

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LAP SHEAR
-423°F



Figure 10-6. Lap Shear Specimens After Test at 21K (-423F)

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LAP SHEAR
350°F



Figure 10-7. Lap Shear Specimens After Test at 450K (350F)

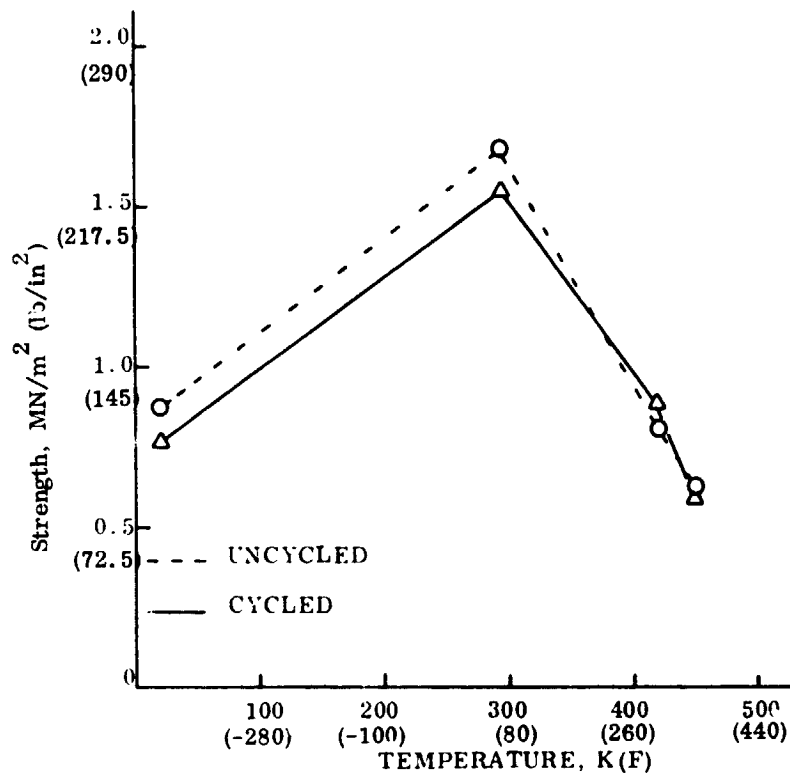


Figure 10-8. Tensile Strength, PPO Foam Bonded with Hysol ADX 394.1 Adhesive

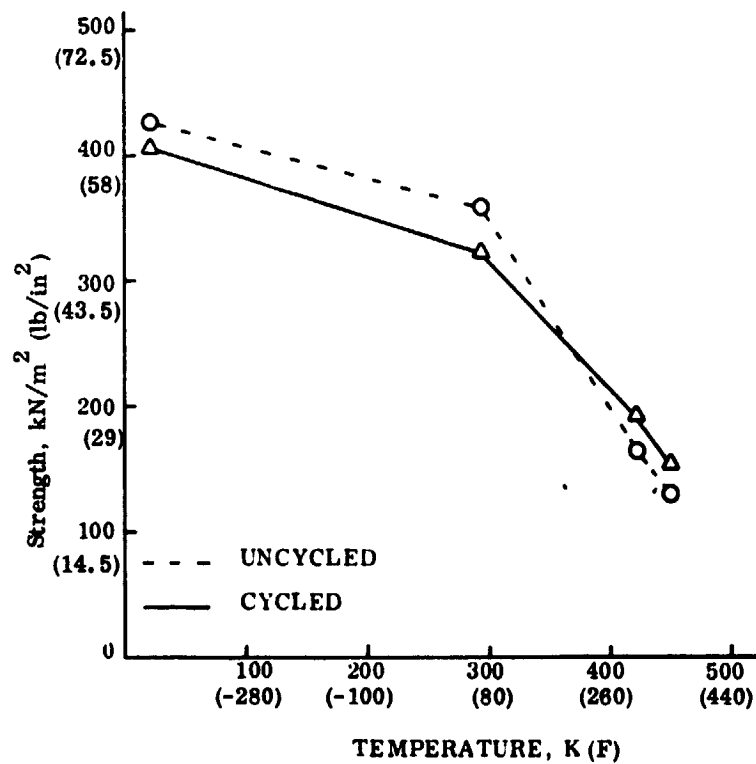


Figure 10-9. Core Shear Strength, PPO Foam Bonded with Hysol ADX 394.1 Adhesive

was no significant change in form or adhesive structural integrity brought about by the thermal cycling. Figures 10 through 13 show cycled face tension and core shear specimens tested at the temperature extremes.

The peel strength of the foam/adhesive specimen is illustrated in Figure 10-14. Specimens after test at cryogenic temperature are shown in Figure 10-15. Although the failures at ambient and cryogenic temperatures appeared to be in the adhesive, there was no marked drop off in peel strength.

Each dogbone specimen was automatically cycled 400 times, at a rate of approximately three cycles per minute, from maximum load to 10 percent of the maximum load (to prevent inadvertent compression) at each of four temperatures. The three specimens in each set begin the test sequence at different points in the cycle to isolate early failures. The dogbones were ultrasonically inspected after each 400 cycle sequence. No disbonds were found in any of the specimens at any point during the test. A typical dogbone monostain test specimen is shown in Figure 10-16.

The following conclusions may be drawn from the results of the Hysol ADX 394-1 adhesive investigation.

1. For all comparable tests, the strengths of the adhesive was significantly greater than that of the polyurethane adhesive at elevated temperatures.
2. Thermal cycling between 21 and 450K (-423 and 350F) resulted in an increase in the tensile lap shear strength of the adhesive and had no significant deleterious effects on other measured values.
3. The 394-1 adhesive survived all of the cyclic loading tests, both longitudinal and transverse, and both before, after and during exposure to repeated severe thermal environments.
4. In all static tests where the foam/adhesive system was evaluated, the foam component suffered the failure except during climbing drum peel at cryogenic and ambient temperatures. Here the measured strength required to peel the specimen was approximately one-third to one-half that of the polyurethane bonded specimens. No criteria for minimum system peel strength has been established.
5. With the possible exception of the peel test results, the Hysol ADX 394-1 adhesive system has demonstrated excellent performance under severe test conditions.

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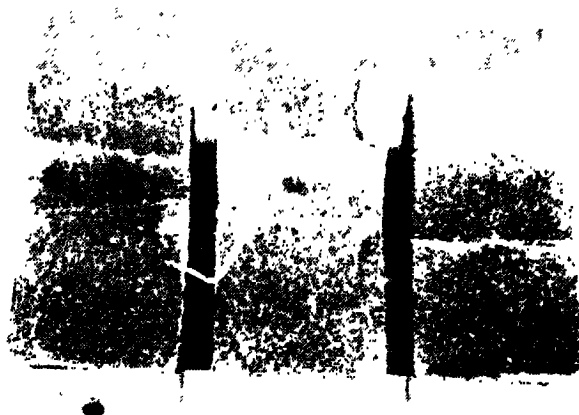
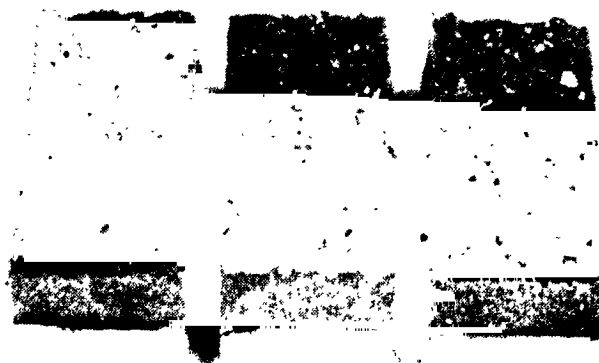


Figure 10-10. Face Tension Specimens After Test at 21K (-423F)

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TENSION
350°F

Figure 10-11. Face Tension Specimens After Test at 450K (350F)

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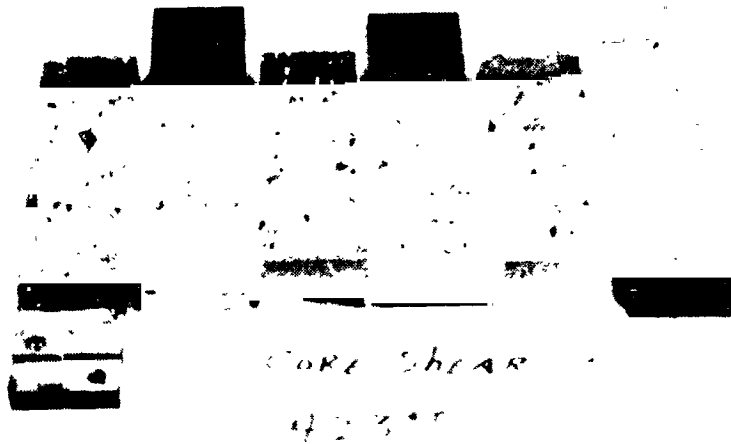


Figure 10-12. Core Shear Specimens After Test at 21K (-423F)

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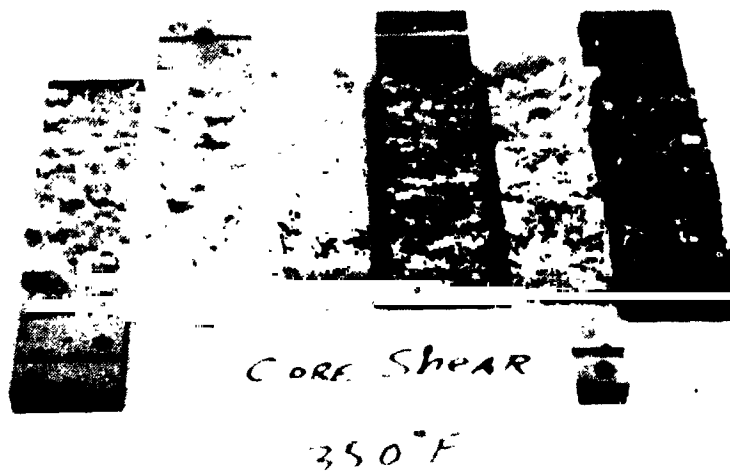


Figure 10-13. Core Shear Specimens After Test at 450K (350F)

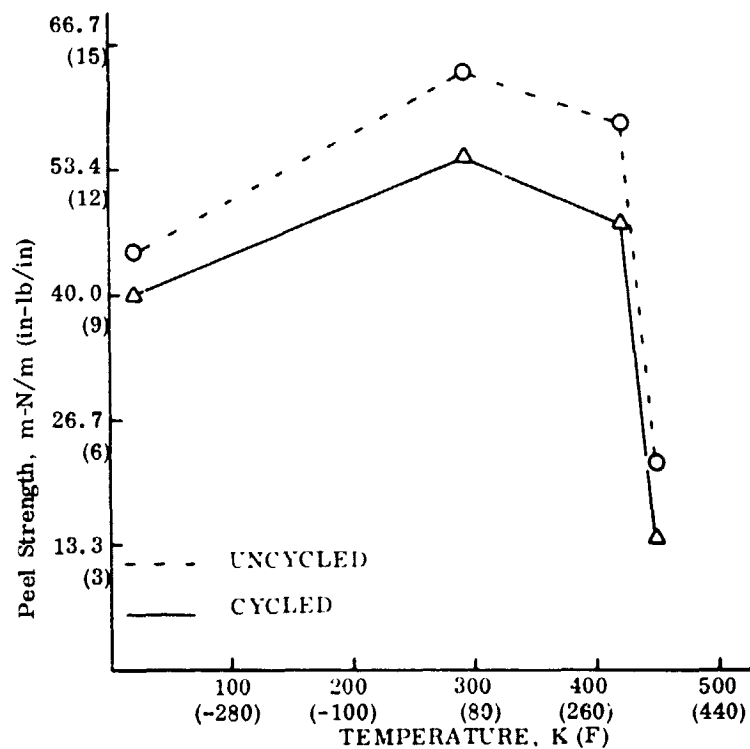


Figure 10-14. Peel Strength, PPO Foam Bonded with Hysol ADX 394.1 Adhesive

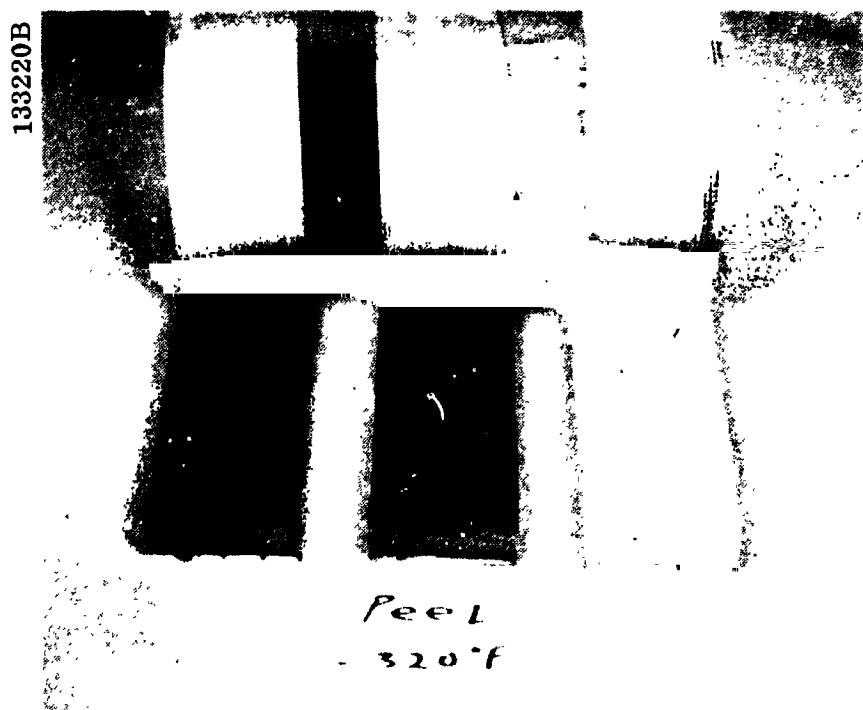


Figure 10-15. Peel Test Specimens After Test at 78K (-320F)

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Figure 10-16. Cyclic Monostrain Test Specimen After Full Series of Tests

CONCLUSIONS AND RECOMMENDATIONS

11.1 CONCLUSIONS

Material Development. Initially forty PPO foam panels of different compositions and densities were inspected and tested. This screening resulted in a materials composition recommendation to the vendor. A preliminary material specification was prepared. The vendor foamed some seventy panels with varying densities and thicknesses of the recommended material. These were generally good quality and were extensively tested and catalogued by Convair. The vendor then initiated a program to set up a semi-automated pilot production line to improve quality control, increase output, and reduce costs. Panels produced and delivered during the following year were hand made using production materials and techniques. Almost without exception these panels were poor quality. Early in 1974 all foam deliveries were stopped. In April 1975 a final ten panel shipment was received. The panels were of good quality, met specification requirements, and had a very low thermal conductivity.

During the course of the PPO foam development, an occasional panel of exceptionally high quality, fine, uniform cell material was obtained. Manufacturing consistently high quality foam is therefore believed to be a matter of obtaining the proper equipment and applying normal chemical process industry quality control standards to the raw materials and processes involved.

Insulation - The material is an excellent gas layer insulation for use with liquid hydrogen. When used with the warm side at room temperature, the thermal conductivity is less than 15 percent greater than gaseous hydrogen. As the warm face approaches LN₂ temperature 78K (140R), the thermal conductivity approaches a value about 40 percent greater than gaseous hydrogen.

The material is sensitive to density gradients parallel to the cell direction. However, when purchased with the density gradients and cell sizes per the specification prepared during this program, a minimum predictable thermal conductivity is assured.

Structural Properties - The anisotropic thermo-mechanical properties make the material ideally suited to cryogenic service. The low strength, low modulus, and high elongation perpendicular to the cells permit the material to move easily with any substrate to which it is bonded. The material is flexible at cryogenic temperature and can be used in an inert atmosphere up to 450K (350F). When service above 367K (200F) is expected, a heat treat should be performed to preshrink and stabilize the material at maximum service temperature in a non-oxidizing atmosphere.

The material is lightweight, 40 kg/cm³ (2.5 lb/ft³), therefore should be usable in many aerospace applications.

Fabrication - PPO foam is an easy material to handle and fabricate. It can be cut with a saw, knife or hot wire. It can be heat formed if desired and also can be easily cold shaped into small radii then bonded with room temperature cure adhesives. Material up to 75 mm thick can be cold shaped into corners where $R/t \geq 1$. Foam-to-foam joints can be compressed and do not require bonding even for a gas layer insulation. If desired, however, large panels can be pre-edge bonded to any handleable size.

Repair - Damaged material is easily removed with a hot wire, scraper, or phenolic cutter. Repair plugs can be compressed and inserted without edge bonding. Removal of the surface adhesive, if grinding is necessary, is the only significant challenge. A panel repaired twice and tested six times over a period of more than a year was degraded thermally only eighteen percent.

11.2 RECOMMENDATIONS

PPO Foam Material Development - PPO foam as it is made today, contains residual unblown particulate matter which apparently has no detrimental affect on its thermo-mechanical properties. It is, however, a potential source of contamination in an internal insulation system. Additional effort is recommended in the raw material mixing and foaming processes to eliminate or minimize this particulate matter.

Cutting and handling also produce debris. It is recommended that PPO foam be used in a "large" liquid hydrogen tank such as a cryogenic component test tank which, over a period of months or years, is used frequently; tanked and detanked. Periodic inspection should be performed on the insulation, discharge lines, and filters to evaluate the extended long term serviceability of PPO foam and the extent of any "debris" problem.

PPO foam is made on essentially the same tooling today as it was 6 years ago. The usable piece size is about 61×71 cm (24 × 28 inches), and the production rate is low. As a result, the foam has limited availability and relatively high price. The cost of phenylene oxide resins is comparable to other plastics used in foams such as polycarbonate and urethane. Styrene, acrylic and PVC are much cheaper. The mixing and foaming process results in the current cost of PPO foam (approximately \$100.00/ft³) compared to urethane which is \$3-\$4/ft³. The low density, handleability and flexibility of PPO foam make it a good candidate sandwich construction and commercial insulating material if the price were reduced. Since its use in aerospace and cryogenic service requires higher quality material, it could be graded, like lumber, and the lower quality material sold at reduced cost to the packaging, appliance, and construction industries. Production of the foam in a reduced cost semi-automated process is considered important to its acceptance and use as a commercial material.

PPO Foam Uses - PPO foam could be used as a gas layer insulation for most liquified gases (except LO_2). Because the thermal conductivity of other gases is lower than hydrogen, the use of PPO foam with these liquids would be comparably more thermally efficient. The required cell size of the PPO foam would in most cases need to be smaller. A program is recommended to continue the effort to reduce the foam cell size and to increase cell uniformity and further improve the density gradient.

PPO foam has unique mechanical properties which makes it an excellent separator material for double walled self evacuated vessels such as pipes and tanks. It is comparable to honeycomb but has a lower strength, lower shear modulus, and finer cell structure which will result in significantly lower loads on the surface bond lines. The foam also has greater capacity to absorb the thermal stresses inherent in a double walled vessel.

Use Expansion Process to Foam Other Plastics - Cell size and uniformity, residual particulate matter, cell friability and cost all need further development. It is possible that plastics other than phenylene oxide could be "foamed" to give the same or even improved properties while solving some of the above mentioned problems. Pursuit of this process with other materials is recommended.

SECTION 12

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APPENDIX A

PPO FOAM PANEL INVENTORIES

1971 Panel Inventory	A-2
1972 Panel Inventory	A-3
1973 Panel Inventory	A-6
1974 Panel Inventory	A-10
1975 Panel Inventory	A-11

1971 PPO Foam Panel Inventory

Identification		Thickness		Size		Density		Blowing Agents(1)	Nucleating Agent(2)
GD/C	TNO	mm	in	cm x cm	in x in	kg/m ³	lb/ft ³		
71-1		75	3	71 x 89	28 x 35	36.4	2.27	DCE	VER 2 phr
-2		75	3	69 x 79	27 x 31			DCE	"
-3		"	"	69 x 76	27 x 30			"	"
-4		"	"	70 x 79	27 1/2 x 31			"	"
-5		"	"	"	"			"	"
-6		"	"	"	"	36.4	2.27	"	"
-7		"	"	"	"			"	"
-8		"	"	"	"			"	"
-9		"	"	"	"			"	"
-10		"	"	"	"			"	"
-11	25-3A	50	2	33 x 43	13 x 17	33.0	2.06	DCE/CNU 1:3	"
-12	24-2	50	2	69 x 79	27 x 31	30.6	1.91	DCE/CNU 1:3	"
-13	-4A	"	"	51 x 74	20 x 29	29.3	1.83	"	"
-14	-4L	"	"	46 x 74	18 x 29	28.8	1.80	"	"
-15	21-7-2A	50	2	46 x 76	18 x 30	42.3	2.64	DCE/CNU 1:3	"
-16	-3A	"	"	"	"	38.6	2.41	"	"
-17	-4A	"	"	"	"	42.1	2.63	DCE/CNU 1:1	"
-18	-5A	"	"	"	"	43.8	2.67	"	"
-19	-6A	"	"	"	"	43.9	2.74	"	"
-20	-8A	"	"	"	"	49.4	3.11	DCE/CNU 3:1	"
-21	-9A	"	"	"	"	47.4	2.96	"	"
-22	-2B	"	"	"	"	45.2	2.82	DCE/CNU 1:3	"
-23	-3B	"	"	"	"	40.0	2.87	"	"
-24	-6B	"	"	"	"	44.9	2.80	DCE/CNU 1:1	"
-25	-7B	"	"	"	"	45.9	2.93	DCE/CNU 3:1	"
-26	-8B	"	"	"	"	47.9	2.99	"	"
-27	1A-9-9-71	50	2	25 x 61	10 x 24	40.2	2.51	DCE/SBP 5:1	"
-28	1B-	"	"	"	"	38.9	2.43	"	"
-29	2' -	"	"	"	"	38.1	2.38	"	"
-30	1-1, 29-5-71	50	2	60 x 78	24 x 31	47.7	2.98	DCE/SBP 5:1	VER 5 phr
-31	2-3/	"	"	48 x 78	19 x 31	41	2.56	"	GEN 2 phr
-32	3-1/	"	"	53 x 80	21 x 32	46.9	2.93	"	VER 5 phr
-33	4-10-71/1-1	50	2	58 x 79	23 x 31	46.3	2.89	DCE	GEN 2 phr
-34	/2-1	"	"	61 x 79	24 x 31	46.4	2.90	"	GEN 5 phr
-35	/3-1	"	"	64 x 81	25 x 32	43.9	2.74	"	VER 5 phr
-36	SP1-2	"	"	53 x 71	21 x 28	37.8	2.36	"	GEN 2 phr
-37	28-9-71/Ex2	50	2	33 x 48	13 x 19			DCE/SBP 5:1	GEN 2 phr
-38	12-10-71/Ex5	"	"	30 x 46	12 x 18			DCE	GEN 2 phr

(1) DCE - Dichloroethane, CNU - Chlorothene Nu (trichloroethane), SBP - Petroleum Ether

(2) VER - vermiculite, GEN - Genitron (azodicarbonamide)

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1972 PPO Foam Panel Inventory

PPO Resin, 61 pph

Blowing Agent: DCE/CNU(1:3), 37 pph

Nucleating Agent: GEN, 2 pph

Open Press

Identifier		Thickness		Size		Density	
GD/C	TNO	mm	in	cm x cm	in x in	kg/m ³	lb/ft ³
72-1	17-11-71/6-2	75	3	60 x 60	23.5 x 23.5	29	1.81
-2	24-11-71/6-1	"	"	"	"	"	"
-3	24-11-71/6-2	"	"	"	"	"	"
-4	24-11-71/6-3	"	"	"	"	"	"
-5	2-12-71/6-9	"	"	"	"	"	"
-6	2-12-71/6-11	"	"	"	"	"	"
-7	18-11-71/1-5	25	1	70 x 70	27.5 x 27.5	33	2.06
-8	23-11-71/1-5	"	"	"	"	"	"
-9	2-12-71/1-16	"	"	"	"	"	"
-10	" /1-17	"	"	"	"	"	"
-11	" /1-18	"	"	"	"	"	"
-12	" /1-21	"	"	"	"	"	"
-13	6-12-71/1-22	"	"	"	"	"	"
-14	" /1-23	"	"	"	"	"	"
-15	" /1-24	"	"	"	"	"	"
-16	" /1-25	"	"	"	"	"	"
-17	" /1-26	"	"	"	"	"	"
-18	" /1-27	"	"	"	"	"	"
-19 ⁽¹⁾	25-3B-71	50	2	33 x 58	13 x 23	"	"
-20 ⁽¹⁾	19-11-71/34 + 39	"	"	61 x 69	24 x 27	45	2.81
-21 ⁽¹⁾	19-11-71/3	"	"	58 x 71	23 x 28	42	2.62
-22 ⁽¹⁾	19-11-71/41 + 44	"	"	58 x 74	23 x 29	42	2.62
-23	3-1-72/4-4	"	"	66 x 76	26 x 30	32	2.00
-24	" /4-5	"	"	"	"	31	1.94
-25	" /4-6	"	"	"	"	29	1.81
-26	" /4-7	"	"	"	"	31	1.94
-27	" /4-8	"	"	"	"	31	1.94
-28	" /4-10	"	"	"	"	31	1.94
-29	18-1-72/5-1	"	"	"	"	41	2.56
-30	" /5-2	"	"	"	"	42	2.62
-31	" /5-3	"	"	"	"	41	2.56
-32	" /5-4	"	"	"	"	44	2.75
-33	" /5-5	"	"	"	"	44	2.75
-34	" /5-6	"	"	"	"	42	2.62
-35	" /5-7	"	"	"	"	42	2.62

1972 PPO Foam Panel Inventory (Cont'd)

Identifier		Thickness		Size		Density	
GD/C	TNO	mm	in	cm × cm	in × in	kg/m ³	lb/ft ³
-36	18-1-72/5-8	50	2	66 × 76	26 × 20	42	2.62
-37	" /5-9	"	"	"	"	42	2.62
-38	" /5-11	"	"	"	"	45	2.81
-39	" /5-12	"	"	"	"	44	2.75
-40	" /5-15	"	"	"	"	42	2.62
-41	5-1-72/7-1	75	3	60 × 60	23.5 × 23.5	40	2.50
-42	" /7-5	"	"	"	"	43	2.69
-43	" /7-6	"	"	"	"	45	2.81
-44	" /7-7	"	"	"	"	43	2.69
-45	" /7-10	"	"	"	"	44	2.75
-46	" /7-11	"	"	"	"	42	2.62
-47	17-1-72/2-5	25	1	66 × 76	26 × 30	44	2.75
-48	" /2-6	"	"	"	"	46	2.87
-49	" /2-7	"	"	"	"	42	2.62
-50	" /2-8	"	"	"	"	46	2.87
-51	" /2-9	"	"	"	"	44	2.75
-52	" /2-10	"	"	"	"	45	2.81
-53	" /2-13	"	"	"	"	46	2.87
-54	" /2-14	"	"	"	"	46	2.87
-55	" /2-15	"	"	"	"	46	2.87
-56	" /2-16	"	"	"	"	45	2.81
-57	" /2-17	"	"	"	"	42	2.62
-58	" /2-18	"	"	"	"	42	2.62
-59	12-1-72/3-1	"	"	"	"	57	3.56
-60	" /3-2	"	"	"	"	60	3.75
-61	" /3-3	"	"	"	"	60	3.75
-62	" /3-4	"	"	"	"	57	3.56
-63	" /3-5	"	"	"	"	58	3.62
-64	" /3-6	"	"	"	"	63	3.94
-65	" /3-7	"	"	"	"	58	3.62
-66	" /3-8	"	"	"	"	61	3.81
-67	" /3-9	"	"	"	"	59	3.69
-68	" /3-10	"	"	"	"	61	3.81
-69	" /3-11	"	"	"	"	59	3.69
-70	" /3-12	"	"	"	"	57	3.56
-71	26-1-72/8-2	75	3	60 × 60	23.5 × 23.5	55	3.44
-72	" /8-3	"	"	"	"	53	3.31
-73	" /8-4	"	"	"	"	54	3.38
-74	" /8-5	"	"	"	"	57	3.56
-75	" /8-6	"	"	"	"	51	3.19
-76	" /8-7	"	"	"	"	52	3.25

1972 PPO Foam Panel Inventory (Cont'd)

Identifier		Thickness		Size		Density	
GD/C	TNO	mm	in	cm × cm	in × in	kg/m ³	lb/ft ³
72-77	14-6-72/9-4	50	2	70 × 70	27.5 × 27.5	53	3.31
-78	9-5	"	"	"	"	54	3.37
-79	9-6	"	"	"	"	53	3.31
-80	9-8	"	"	"	"	53	3.31
-81	9-9	"	"	"	"	54	3.37
-82	9-12	"	"	"	"	54	3.37
-83	13-6-72/8-10	75	3	60 × 60	23.6 × 23.6	52	3.25
-84	8-11	"	"	"	"	55	3.44
-85	8-12	"	"	"	"	52	3.25
-86	8-16	"	"	"	"	53	3.31
-87	8-18	"	"	"	"	53	3.31
-88	8-19	"	"	"	"	53	3.12
-89	10-9	45	1 3/4	40 × 40	15.8 × 15.8	62	3.87
-90	10-13	50	2	"	"	70	4.37
-91	6210-45	"	"	30D	11.7D	45	2.81
-92	2209-31	"	"	25D	9.8D	45	2.81
-93	21-10-7153	140	5 1/2	30 × 34	12 × 13.5	33	2.06

(1) New Screening Panels

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1973 PPO Foam Panel Inventory

Unless otherwise indicated:

Blowing agent, DCE/CNU 3:1
Nucleating agent, Vermiculite
Thickness, 50 mm (2 in.)

Two mill sheets
Open press
Shipped with protective paper

ID/C	Identification	Size		Density			X-ray	Comments	Date Rec'd.
		cm x cm	in. x in.	kg/m ³	lb/ft ³	Paper Off kg/m ³			
73-1	051272/10-6	60x60	23.6x23.6	93	5.81	86	x	Shipped w/o paper, cut up and inspected	Jan. 2
-2	/10-8			93	5.81	85	x		
-3	111272/10-11			72	4.50	64	x		
-4	/10-12			75	4.68	68	x		
-5	/10-13			75	4.68	67	x		
-6	/10-14			72	4.50	66	x		
-7	/10-15			71	4.43	64	x		
-8	/10-17			74	4.62	66	x		
-9	301172/11-1	50x50	19.7x19.7	105	6.56	93	x		
-10	/11-3			102	6.37	93	x		
-11	/11-4			97	6.06	89	x		
-12	11-6			100	6.25	93	x		
-123	15-3-73-2	60x70	23.5x27.5	42.5	2.65			Closed press	Apr. 16
-124	15-3-73-4	60x70	23.5x27.5	44	2.75			Closed press	Apr. 16
-125	EX3-51173	63.5x73.7	25x29	46	2.87		x	Shipped w/o paper, 3 sheet extruded ρ grad and ΔP tests	Nov. 30
-126	15-3-73-1	60x67.3	23.5x26.5	38	2.37		x	1 sheet milled	Nov. 30
-127	121073-15	63.5x73.7	25x29	40	2.50		..	Shipped w/o paper, 3 sheet milled ρ grad and ΔP tests, thickness 12 cm (4.7 in.)	Nov. 30

1973 PPO Foam Panel Inventory (Continued)

Size: 70 x 80 cm (27.6 x 31.5 in.)

Thickness: 50 mm (2 in.)

Identification		Density		Comments	Date Rec'd.
GD/C	TNO	kg/m ³	lb/ft ³		
73-13	181272/5-5	43	2.69	Stock	Jan. 22
-14	5-8	43	2.69		
-15	5-9	42	2.62		
-16	5-11	43	2.69		
-17	5-12	43	2.69		
-18	5-13	43	2.69		
-19	5-15	42	2.62		
-20	201272/5-16	42	2.62		
-21	5-17	41	2.56		
-22	5-19	41	2.56		
-23	5-20	41	2.56		
-24	5-21	41	2.56		
-25	5-22	40.5	2.53		
-26	5-23	39.5	2.47		
-27	5-24	41	2.56		
-28	5-25	41	2.56		
-29	5-26	41.5	2.59		
-30	5-27	41	2.56		
-31	5-28	43	2.69		
-32	5-29	42.5	2.65		
-33	211272/5-32	42	2.62		
-34	5-33	41	2.56		
-35	5-35	41.5	2.59		
-36	5-36	42	2.62		
-37	5-38	43	2.69		
-38	5-39	42	2.62		
-39	5-40	42	2.62		
-40	5-41	43	2.69		
-41	5-42	43	2.69		
-42	5-43	43	2.69		
-43	5-44	42	2.62		
-44	5-46	42	2.62		
-45	5-47	43	2.69		
-46	020173/5-48	44	2.75		
-47	5-50	41	2.56		
-48	5-51	42	2.62	X-rayed, to Plt. 19 w/ tanks	
-49	5-52	42	2.62		
-50	5-53	41.5	2.59		

1973 PPO Foam Panel Inventory (Continued)

Size: 70 × 80 cm (27.6 × 31.5 in.)

Thickness: 50 mm (2 in.)

Identification		Density		Comments	Date Rec'd.
GD/C	TNO	kg/m ³	lb/ft ³		
73-51	020173/5-54	41	2.56	Large scale bonded panel	Jan. 22
-52	5-56	↓	↓	" " " "	
-53	5-57	↓	↓	X-rayed, Mat'l.Res.Lab Insulation	↓
-54	5-58	↓	↓	↓	
-55	5-59	41.5	2.59	↓	
-56	5-60	41	2.56	Large scale bonded panel	
-57	5-61	41.5	2.59	↓	
-58	5-62	39.5	2.47	↓	
-59	5-63	41	2.56	↓	
-60	5-64	42	2.62	↓	
-61	5-66	41	2.56	↓	
-62	5-67	41.5	2.59	↓	
-63	5-68	41.5	2.59	↓	
-64	5-69	42	2.62	↓	
-65	5-70	↓	↓	↓	
-66	5-71	↓	↓	↓	
-67	5-72	↓	↓	X-rayed	
-68	5-73	↓	↓	Joint Test	
-69	5-74	41.5	2.59	" "	
-70	5-76	42	2.62	Deflection Test	
-71	5-77	↓	↓	Repair Material	
-72	5-78	↓	↓	Joint Test	
-73	030173/5-79	↓	↓	" "	↓
-74	040173/5-80	↓	↓	Stock	
-75	5-81	43.5	2.72	↓	
-76	5-82	44	2.75	↓	
-77	5-84	41.5	2.59	↓	
-78	5-86	42.5	2.65	↓	
-79	5-87	42	2.62	↓	
-80	5-88	42	2.62	↓	
-81	5-90	44	2.75	↓	
-82	5-91	41	2.56	↓	
-83	5-92	42	2.62	↓	
-84	5-93	43	2.69	↓	
-85	5-94	43.5	2.72	↓	
-86	5-96	41.5	2.59	↓	
-87	5-97	43.5	2.72	↓	
-88	5-98	43	2.69	↓	

1973 PPO Foam Panel Inventory (Continued)

Size: 70 · 80 cm (27.6 · 31.5 in.)

Thickness: 50 mm (2 in.)

Identification		Density		Comments	Date Rec'd.
GD/C	TNO	Vendor Data			
		kg/m ³	lb/ft ³		
73-89	040173/5-99	44	2.75	Stock	Feb. 12
-90	5-100	42.5	2.65		
-91	5-101	42	2.62		
-92	5-102	42	2.62		
-93	5-103	41.5	2.59		
-94	5-106	41.5	2.59		
-95	5-107	42.5	2.65		
-96	5-108	40.5	2.53		
-97	5-109	41	2.56		
-98	5-110	41	2.56		
-99	5-111	42	2.62		
-100	5-112	41.5	2.59		
-101	160173/5-113	42	2.62		
-102	5-114	42	2.62		
-103	170173/5-116	42.5	2.65		
-104	5-118	41.5	2.59		
-105	5-119	43	2.69		
-106	5-120	42	2.62		
-107	5-121	40	2.50		
-108	5-122	42	2.62		
-109	5-123	41	2.56		
-110	5-124	40	2.50		
-111	5-127	41	2.56		
-112	5-128	41	2.56		
-113	5-130	41.5	2.59		
-114	5-131	42	2.62		
-115	5-132	42	2.62		
-116	5-133	43	2.69		
-117	5-134	42	2.62		
-118	5-136	42	2.62		
-119	5-137	43	2.69		
-120	5-138	42.5	2.65		
-121	5-139	42.5	2.65		
-122	5-140	43	2.69		

1974 PPO Foam Panel Inventory

Date Received: 5 February 1974

Size 61.5 71.4 cm (25.4 x 29.3 in.)

Identification		Thickness		Density		X-ray	Comments
GD/C	TNO	mm	in.	kg/m ³	pcf		
7-1	12173-10	53.3	2.1	42.5	2.7	x	Extruded, 2 MS DCE, 5 phr
2	12173-11	53.3	2.1	43.3	2.7	x	Extruded, 2 MS DCE, 5 phr
3	141273-22	13.2	1.7	38.5	2.4	x	Extruded, 2 MS DCE, 5 phr
4	111273-1	13.2	1.7	44.5	2.8	x	Extruded, 2 MS DCE, 5 phr
5	111273-18	15.7	1.8	43.5	2.7		Extruded, 2 MS DCE, 5 phr
6	111273-3	45.7	1.8	45.0	2.8		Extruded, 2 MS DCE, 5 phr
7	141273-16	53.3	2.1	42.0	2.6		Extruded, 2 MS DCE, 5 phr
8	141273-20		2.1	41.0	2.6		Extruded, 2 MS DCE, 5 phr
9	080174-3		2.1	42.0	2.6	x	Milled, 1 MS DCE/CNU, 3 phr
10	141273-17		2.1	43.5	2.7		Extruded, 2 MS DCE 5 phr
11	111273-5		2.1	40.0	2.5	x	Extruded, 2 MS DCE/CNU, 5 phr
12	111273-4		1.8	42.0	2.6	x	Extruded, 2 MS DCE/CNU, 5 phr
13	141273-24		2.1	39.0	2.4		Extruded, 2 MS DCE
14	141273-23		2.1	38.0	2.4		Extruded, 2 MS DCE
15	080174-1		2.1	43.0	2.7	x	Milled, 1 MS DCE/CNU, 3 phr
16	080174-2		2.1	43.5	2.7	x	Milled, 1 MS DCE/CNU, 3 phr
17	090174-26	58.4	2.3	119.0	7.4		Milled, 3 MS DCE, 5 phr
18	090174-24	53.3	2.1	132.0	8.2		Milled, 3 MS DCE, 5 phr
19	090174-30		2.1	128.0	8.0		Milled, 3 MS DCE, 5 phr
20	090174-23		2.1	126.0	7.9	x	Milled, 3 MS DCE, 5 phr
21	080174-12		2.1	41.5	2.8		Milled, 1 MS DCE/CNU, 3 phr
22	080174-32		2.1	130.0	8.1		Milled, 3 MS DCE, 5 phr
23	080174-7		2.1	42.0	2.6		Milled, 1 MS DCE/CNU, 3 phr
24	080174-10		2.1	45.5	2.8		Milled, 1 MS DCE/CNU, 3 phr
25	080174-6		2.1	45.5	2.8		Milled, 1 MS DCE/CNU, 3 phr
26	12173-6		2.1	42.5	2.7	x	Extruded, 2 MS DCE/CNU, 5 phr
27	040174-34		2.1	110.0	6.9	x	Extruded, 7 MS DCE, 5 phr
28	040174-35		2.1	113.0	7.1		Extruded, 7 MS DCE, 5 phr
29	090174-29		2.1	128.0	8.0		Milled, 3 MS DCE, 5 phr
30	080174-8		2.1	41.5	2.6		Milled, 1 MS DCE/CNU, 3 phr
31	110174-42	76.2	3.0	43.5	2.7	x	Milled, 2 MS DCE/CNU, 3 phr
32	110174-34		3.0	44.0	2.7	x	Milled, 2 MS DCE/CNU, 3 phr
33	110174-36		3.0	43.0	2.7	x	Milled, 2 MS DCE/CNU, 3 phr
34	110174-41		3.0	45.5	2.8	x	Milled, 2 MS DCE/CNU, 3 phr
35	110174-35		3.0	43.0	2.7	x	Milled, 2 MS DCE/CNU, 3 phr
36	040174-32		3.0	42.0	2.6	x	Extruded, 3 MS DCE, 5 phr
37	040174-30		3.0	42.0	2.6	x	Extruded, 3 MS DCE, 5 phr
38	110174-40		3.0	46.0	2.9	x	Milled, 2 MS DCE/CNU, 3 phr
39	110174-39		3.0	45.0	2.8	x	Milled, 2 MS DCE/CNU, 3 phr
40	040174-28		3.0	43.0	2.7	x	Extruded, 3 MS DCE, 5 phr
41	040174-26	68.6	2.7	42.0	2.6	x	Extruded, 3 MS DCE, 5 phr
42	040174-31	78.7	3.1	38.0	2.4	x	Extruded, 3 MS DCE, 5 phr
43	040174-29	76.2	3.0	39.0	2.4	x	Extruded, 3 MS DCE, 5 phr
44	110174-37	76.2	3.0	45.0	2.8	x	Milled, 2 MS DCE/CNU, 3 phr
45	090174-21	185.4	7.3	48.0	3.0		Milled, 4 MS DCE/CNU, 3 phr
46	070174-13		7.2	47.0	2.9	x	Milled, 4 MS DCE/CNU, 3 phr
47	090174-19		7.3	49.0	3.1		Milled, 4 MS DCE/CNU, 3 phr
48	090174-22		7.3	48.0	3.0		Milled, 4 MS DCE/CNU, 3 phr
49	070174-14		7.3	47.0	2.9		Milled, 4 MS DCE/CNU, 3 phr
50	090174-20		7.3	48.0	3.0		Milled, 4 MS DCE/CNU, 3 phr

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1975 PPO FOAM PANEL INVENTORY

PPO Resin, 71 pph

Blowing agent: DCE/CN¹ (3:1), 27 pph

Nucleating agent: VER, 2pph

One mill sheet

Closed Press

Date received: 25 April 1975

Shipped w/ protective paper

Thickness: 50 mm (2 in.)

Size: 60 × 70 cm (23.6 × 27.6 in.)

Identification		Density				x-ray	Disposition
		Vendor Data		Paper Off			
GD/C	TNO	kg/m ³	lb/ft ³	kg/m ³	lb/ft ³		
75-1	140475-1	42.9	2.68	40.6	2.53	x	Stock
-2	-3	44.3	2.77	42.8	2.67	x	Stock
-3	-4	42.7	2.67	41.6	2.60	x	Four strain isolator sheets, .150"
-4	-5	43.7	2.73	42.5	2.65	x	Stock
-5	-6	42.5	2.65	41.2	2.57	x	Stock
-6	-7	42.7	2.67	40.9	2.55	x	Stock
-7	-8	43.8	2.73	42.0	2.62	x	2 grad.
-8	-9	42.5	2.65	39.7	2.48	x	Vendor Q.C. panel Cut into 27 2 grad pcs.
-9	-11	41.2	2.57	40.1	2.50	x	2 grad.
-10	-12	42.5	2.65	41.0	2.56	x	Thermal conductivity

APPENDIX B

TEST PROCEDURE PPO FOAM

THERMAL CONDUCTIVITY

APPENDIX B

TEST PROCEDURE PPO FOAM THERMAL CONDUCTIVITY

1. Install test specimen in horizontal position. Check T/C and heater resistances.
2. Record T/C's 1-11, test heater volts and amps, guard heater volts and amps, and tank absolute pressure (or gauge pressure plus ambient pressure).
3. Put 20 volts on each of the heaters.
4. Chill the tank slowly with GH₂ and fill slowly with LH₂. Set heater voltages to maintain T/C's 9 and 10 at -100°F (7.70 mv*).
5. Stabilize tank pressure at 15.5 ± 0.3 psia.
6. Adjust test heater power to set T/C 1 at $180 \pm 10^{\circ}\text{F}$. Maintain ΔT between T/C's 9 and 10 less than 2°F (~ 0.08 mv). Stabilize. Take the specimen through all of the equilibrium points given below.

	T/C 1		T/C's 9 and 10	
	<u>$^{\circ}\text{F}$</u>	<u>mv</u>	<u>$^{\circ}\text{F}$</u>	<u>mv</u>
(1)	180 ± 10	13.85 (14.22 - 13.49)	± 2	± 0.08
(2)	100 ± 10	10.99 (11.34 - 10.65)	± 2	± 0.07
(3)	0 ± 10	7.69 (8.01 - 7.38)	± 2	± 0.07
(4)	-100 ± 10	4.74 (5.02 - 4.47)	± 2	± 0.06
(5)	-200 ± 10	2.25 (2.47 - 2.02)	± 2	± 0.05
(6)	-300 ± 10	0.31 (0.48 - 0.16)	± 2	± 0.04
(7)	-100 ± 10	4.74 (5.02 - 4.47)	± 2	± 0.06

7. Slowly pressurize the tank to 40 psia (≤ 1 psi per minute). Do not change test section heater power setting. Adjust guard section power to maintain ΔT between T/C 9 and 10 $\leq 1^{\circ}\text{F}$ (~ 0.06 mv). Stabilize.
8. Increase the test heater power (if necessary) to bring T/C 1 up to -100°F . Maintain $\Delta T \leq 1^{\circ}\text{F}$. Stabilize.
9. Depressurize the tank slowly to 15.5 ± 0.03 psia.

* All mv values are for Chromel-Constantan T/C's (LN₂ ref.)

10. Reorient the specimen to the vertical position.
11. Repeat steps 6 through 9.
12. Adjust the test heater power to set T/C 1 at $-100 \pm 10^{\circ}\text{F}$. Maintain ΔT between T/C's 9 and 10 less than 2°F ($\sim 0.03 \text{ mv}$). Stabilize.
13. Set the Dymec on 10 sec scan. Pressurize the tank to 40 psia at a constant rate between 3 and 5 psi/sec.
14. Five minutes after reaching 40 psia, change back to one minute scans. Do not change the test section heater power setting. Adjust guard section power to maintain ΔT between T/C's 9 and 10 $\leq 1^{\circ}\text{F}$ ($\sim 0.06 \text{ mv}$). Stabilize.
15. Increase the test heater power (if necessary) to bring T/C 1 up to -100°F . Maintain $\Delta T \leq 1^{\circ}\text{F}$. Stabilize.
16. Depressurize the tank slowly to $15.5 \pm 0.3 \text{ psia}$.
17. Repeat Step 12.
18. Terminate the test.

APPENDIX C

**SPECIFICATION FOR
POLYPHENYLENE OXIDE (PPO) FOAM
FLEXIBLE ANISOTROPIC, OPEN
CELL INSULATION MATERIAL**

GENERAL DYNAMICS Convair Division CODE IDENT NO. 14170				SPECIFICATION NO. 0-06212		REV	
				DATE 1 May 1975			
				CONTRACT NO. NAS 8-27566			
Number of Pages <u>17</u>							
Polyphenylene Oxide (PPO) Foam Flexible Anisotropic, Open Cell Insulation Material, Specification for							
PREPARED BY <u>G. B. Yates</u> G. B. Yates CHECKED BY <u>L. R. Hume</u>				APPROVED BY <u>H. H. Ross</u>			
SPECIFICATION CHANGE NOTICES—INCORPORATED IN SPEC REVISIONS							
SCN NO(S)	INCRP BY	CHECK BY	APPROVED BY	DATE	SPEC REV	REVISION PACKAGE NO.	RELEASE
INITIAL PACKAGE NO.				RELEASE	ECC TYPE	DISTR CODE	
6151-643-4-486				7/10	20	NONE	

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1. SCOPE

1.1 Scope. This material specification establishes the requirements for flexible anisotropic polyphenylene oxide (PPO) foam open cell insulation material.

1.2 Classification. The PPO foam shall be classified in accordance with cell edge surface condition as follows (see also 6.4.4):

Class I - delivered with the press platen protective coating sheets removed.

Class II - delivered with the press platen protective coating sheets attached.

2. APPLICABLE DOCUMENTS

2.1 Standards. The following documents of the issue in effect on the date of Convair's request for quotation form a part of this specification.

Military MIL-STD-105

Sampling Procedures and
Tables for Inspection by
Attributes

ASTM C 273-61

Shear Test in Flatwise Plane
of Sandwich Constructions

C 297-71

Tension Test in Flatwise Plane
of Sandwich Construction

NAS 850

General Packaging Standard

3. REQUIREMENTS

3.1 Qualification. The material furnished under this specification shall be a product which has passed the qualification test specified herein and the formulations (see 3.2.1) have been approved by General Dynamics/Convair.

3.2 Materials. The material shall consist of pure PPO resin combined for manufacturing purposes with a solvent (plasticizer/blowing agent) and a nucleating agent.

3.2.1 Components. The material described herein shall consist of:

- a. Polyphenylene oxide powder grade C1000, PR 100 natural.
- b. Solvents. The powder is mixed with a suitable plasticizing/blowing agent for sheet forming and expansion. The supplier shall stipulate the chemical names and the parts per hundred weight resin of each solvent used.
- c. Nucleating agent. A suitable nucleating agent is included in the raw material mixture. The supplier shall stipulate the chemical name and the parts per hundred weight resin of the nucleating agent.

3.2.2 Class I material. The class I material shall be furnished as flat sheets with the press platen protective covers (paper, plastic, aluminum) removed. Each sheet shall be flat and square within tolerances specified in 3.4.1. Removal of the protective covers shall include not more than 2.0 mm of parent material on each surface.

3.2.3 Class II material. The class II material, 6.4.4, shall be furnished as flat sheets with the press platen protective covers (paper, plastic or aluminum) attached as blown. Each sheet shall be flat and square within the tolerances specified in 3.4.2.

3.3 Storage Life

3.3.1 Class I material. The storage life of Class I material shall be unlimited when stored in a clean, lint, dust and dirt free environment below 27C and 80% relative humidity. Class I material shall not be exposed to ultraviolet light during storage. If stored more than 24 months, mechanical properties per 3.6 shall be verified before use.

3.3.2 Class II material. The storage life of Class II material shall be unlimited when stored in a clean, lint, dust and dirt free environment below 27C and 80% relative humidity.

3.4 Dimensions and Tolerances

3.4.1 Class I materials

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3.4.1.1 Length and width. The average length and width of each individual foam panel shall be within ± 5 mm of the specified values. The variation in length and width of each individual panel shall be within ± 5 mm of the average values for that panel.

3.4.1.2 Thickness. The average thickness of each individual foam panel shall be within $\pm .25$ mm of the specified value.

3.4.2 Class II material.

3.4.2.1 Length and width. The average length and width of each individual foam panel shall be within ± 5 mm of the specified values. The variation in length and width of each individual panel shall be within ± 5 mm of the average values for that panel.

3.4.2.2 Thickness. The average thickness of each individual foam panel shall be within ± 2.5 mm of the specified value. The panel with the protective coating sheets attached shall be flat to within ± 1 mm.

3.5 Physical Properties. The physical properties as specified herein apply to measurements made to Class I material only.

3.5.1 Density. Bulk density is herein defined as the ratio of the mass of a trimmed foam panel in kilograms to its bulk volume in cubic meters. The value specified in an order is for the Class I foam panel. The measured bulk density of each individual foam panel shall be within ± 5 percent of the specified value in 6.4.4.

3.5.2 Density Gradient. The measured bulk density variations within a foam panel shall be limited to ± 5 percent of the specified value in 6.4.4 perpendicular to the fiber direction. The measured bulk density of the inner one-third of a foam panel parallel to the fiber direction shall be not less than 90 percent of the density of the overall panel.

3.5.3 Cell size. There shall be no voids in the foam having a diameter greater than 1.5 mm. The average diameter of the 10 largest cells in any 4 cm^2 area shall not exceed 0.5 mm. Diameter is the longest dimension across any irregular cell perpendicular to the fiber direction.

3.5.4 Flow conductance. The gas flow conductance through the foam perpendicular to the fiber direction shall be less than the conductance parallel to the fiber direction. Conductance shall be defined as the gas mass flow rate per unit of surface area per unit of thickness for a given pressure differential.

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3.6 Mechanical Properties

3.6.1 Tensile Modulus. The room temperature tensile modulus of elasticity in the fiber direction shall be greater than 800 Kg/cm^2

3.6.2 Shear Modulus. The room temperature effective core shear modulus of elasticity in the fiber direction shall be TBD.

3.7 Workmanship. The material shall be manufactured in accordance with first grade practice to produce material of uniform quality free from defects of any character. The foam surface perpendicular to the fiber direction when cut to the Class I configuration shall be smooth with no ragged or torn cell edges.

4. QUALITY ASSURANCE

4.1 Responsibility for inspection.

4.1.1 Class I materials. Unless otherwise specified in the contract or order, the supplier shall be responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may use his own facilities or any commercial laboratory acceptable to GD/Convair. GD/Convair reserves the right to perform any or all of the inspections set forth herein where such inspections are deemed necessary to assure that the material to be furnished conforms to the prescribed requirements.

4.1.2 Class II materials. When delivery of Class II materials is specified, the supplier shall cut-off the protective cover only from those panels required to meet the test requirements of 4.5.

4.2 Inspection records. Inspection records of examinations and tests shall be kept complete and available to GD/Convair. These records shall contain all data necessary to determine compliance with the requirements of this specification. Density and density gradient specimens and all measurements shall be shipped with each order.

4.3 Classification of inspection. The inspection of the material shall be classified as follows:

- a. Qualification tests
- b. Acceptance tests

4.3.1 Qualification tests. Before any material is approved for procurement in accordance with this specification, qualification tests shall have been performed to insure that material meets all requirements of this specification. Testing shall be as specified in 4.5.

Qualification shall be performed on an initial procurement from any vendor with a stipulated set of components per 3.2.1. Any variation in components or component quantities shall require requalification of the material per 6.4.2.

4.3.2 Acceptance tests. Acceptance tests for each lot of material shall be from a representative sampling per 4.4 and test methods per 4.5.

4.4 Sampling. The controls used during manufacture shall assure that each lot of material offered for delivery meets the requirements specified herein. Test per 4.5.2, 4.5.3.1, and 4.5.3.3.1 shall be performed on each individual panel. Tests per 4.5.3.2, and 4.5.3.3.2 shall be performed on every tenth manufactured panel or one per order which ever is greater. Tests per 4.5.3.4 and 4.5.4 shall be performed on every twenty-fifth manufactured panel or one per order which ever is greater.

4.5 Test Methods.

4.5.1 Measurement Accuracy. The absolute accuracy of the measurements made in compliance with this specification shall be as follows:

- a. Length and width, ± 1 mm
- b. Thickness, ± 0.05 mm
- c. Weight, ± 5 gm
- d. Flow rate, ± 5 percent
- e. Pressure, ± 5 percent

4.5.2 Dimensions. Dimension measurements shall be recorded in the format of Table 1.

4.5.2.1 Length and width. A minimum of three measurements shall be made of the width and three of the length of each edge trimmed PPO foam panel. Both the arithmetic average and individual measurements shall be evaluated for conformance with 3.4.1.1.

4.5.2.2 Thickness. A minimum of five measurements shall be made of the thickness of each PPO foam panel. The arithmetic average of the measurements shall be evaluated for conformance with 3.4.1.2.

4.5.3 Physical Properties.4.5.3.1 Density.Class I material.

The average dimensions obtained in 4.5.2 together with the measured weight of each panel shall be used to calculate the bulk density. The value obtained shall be evaluated for conformance with 3.5.1. The density measurements shall be recorded in the format of Table 1.

Class II material.

The average dimension obtained in 4.5.2 together with the measured weight of each panel shall be recorded in the format of Table 1.

4.5.3.2 Density gradient. Every tenth PPO foam panel manufactured for a given order shall be investigated for density gradients within the panel. The protective cover shall first be cut off of the Class I configuration per 3.2.2. The panel shall then be cut into three equal pieces and identified as k=1, 3 (see Figure 1). Immediately after cutting, the proper identification number shall be inscribed in ink on the upper left-hand corner of the edge of each piece cut.

Each piece shall be weighed and measured and the density shall be calculated using the same procedure per 4.5.3.1. The calculated values shall be evaluated for conformance with 3.5.2.

Finally each of the three pieces shall be sliced into tiles, as shown in Figure 1, and identified as i, j, k_{i, j, k=1, 3} (see Figure 1). Densities shall be calculated as described above and the values shall be evaluated for conformance with 3.5.2.

The density gradient dimensions, weights, and sub panel densities shall be recorded in the format of Table 2.

4.5.3.3 Cell size.

4.5.3.3.1 X-ray. An X-ray exposure shall be made of each individual edge trimmed PPO foam panel. Multiple exposures may be made if necessary to cover the entire panel. X-ray parameters used successfully by GD/Convair are listed below:

- a. X-ray tube, NORELCO MG 50 with beryllium window or equivalent
- b. focal spot, 15 mm

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|------------------|--|
| c. power, | 10 KV at 15 ma, or lower |
| d. film, | Eastman Kodak Type T medium grain or equivalent |
| e. exposure time | Vary with equipment and film to give maximum density gradient contrast |

The exposure(s) shall be examined and indicated voids or high density resin concentrations evaluated.

4.5.3.3.2 Diameter. Cell edges shall first be examined for conformance with 3.7. The cell diameters shall be measured in a 2 cm square on six of the density gradient sub panels in 4.5.3.2. The measurements shall be made on two k = 1 panels, two k = 2 panels, and two k = 3 panels. Selection of the sub panels for measurement shall be based on visual inspection and examination of the X-ray film for areas with the largest cells.

Cell sizes and voids shall be evaluated for conformance with 3.5.3.

4.5.3.1 Flow conduction. Every twenty-fifth panel manufactured for a given order shall be investigated to determine flow conductance and mechanical strength. The protective cover shall first be cut off to the Class I configuration per 3.2.2. The panel shall then be cut to produce a total of 12 test specimens, Figure 2. The dimensions of each piece shall be determined per 4.5.1. The rectangular pieces cut from quadrants 1 and 4 shall be used for flow conductance measurements.

Flow conductance measurements shall be made using an apparatus such as illustrated in Figure 3. The two pieces shall be used first for measurements perpendicular to the cell axes then trimmed for measurements parallel to the cell axes. Each piece shall be installed in the apparatus such that leakage around the edge is negligible. Dry room temperature nitrogen gas shall be flowed through the piece and both gas mass flow rate and differential pressure shall be determined. The flow shall be set and measured at five points corresponding to differential pressures of 2.0, 4.0, 6.0, 8.0 and 10.0 kN/m². The flow conductance and conductance ratios shall be calculated and tabulated in the format of Table 3. The values shall be evaluated for conformance with 3.5.4.

4.5.4 Mechanical properties test. Mechanical strength measurements shall be made on a standard load testing machine. The specimens each shall be tested at 75 ± 5° F. Specimens from every twenty fifth manufactured panel of a given order shall be tested, 4.5.3.4. The average results shall pass the specified requirements per 3.6. The test methods shall be as follows:

- a. Tensile strength: ASTM C 297-61
- b. Shear strength: ASTM C 273-61

4.5.1.1 Tensile tests. The six square pieces, three each from quadrants 1 and 4, Figure 2, shall be bonded to plates and subjected to tensile loading until the proportional limit of the material is determined. Any failures must occur in the foam rather than in the bond line. The modulus shall be calculated, the six data points arithmetically averaged, and the data tabulated in the format of Table 4. The results shall be evaluated for conformance with 3.6.1.

4.5.1.2 Shear tests. The four rectangular pieces, two each from quadrants 2 and 3, Figure 2, shall be bonded to plates and subjected to shear loading in the 2t, "length" direction until the proportional limit of the material is reached. Any failures must occur in the foam rather than at the bond line. The modulus shall be calculated, the four data points arithmetically averaged, and the data tabulated in the format of Table 4. The results shall be evaluated for conformance with 3.6.2.

5. PREPARATION FOR DELIVERY

5.1 Preservation and packaging. The material shall be in suitable containers in quantities as specified on the purchase order. All materials shall be packaged to insure protection from physical damage during handling, shipping and storage.

5.2 Packing. The material shall be packed in shipping containers of a type which shall adequately protect the material during normal handling and meet the minimum packing requirements of common carriers for acceptance and safe transportation at the lowest rate to the point of delivery.

5.3 Marking for shipment. Each unit and intermediate container shall be identified with a suitable label or tag with information as follows:

- a. GD/Convair 0-06212 and applicable material identification dash number.
- b. Manufacturer and product designation.
- c. Lot number and date of manufacture.
- d. Purchase order or contract number.

In addition, shipping containers shall be marked with the address of the procuring agency as indicated on the purchase order or contract.

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5.4 Documentation and reporting. Inspection and quality assurance test data shall be documented and reported as described herein.

5.4.1 Documentation. All manufactured panels shall be numbered prior to inspection and testing. All X-ray exposure prints and test data shall be referenced to a panel identification number. Dimensional averages and variations as well as the calculated bulk density for every panel produced shall be recorded in a table similar to Table 1.

Density gradient data, dimensions, weights and densities shall be recorded in a table similar to Table 2. Flow conductance test data shall be recorded in a table similar to Table 3. Shear and tensile strength data shall be recorded in a table similar to Table 4.

5.4.2 Reporting. Copies of all data sheets and X-ray prints shall be shipped simultaneously with the panels. All samples tested per 4.5.3.2 and 4.5.3.4 shall be boxed and shipped simultaneously with the panels. A list of all the panels rejected and the reason for rejection shall be shipped simultaneously with the panels.

6. NOTES

6.1 Intended use. The material described by this specification is intended for use as internal thermal insulation for liquid hydrogen tanks.

6.2 Ordering information If certification is required, it should be stated on the purchase order that two copies of the certification should accompany each shipment sent to GD/Convair. The following information should be included on the purchase order:

- a. GD/Convair 0-00212 and appropriate material identification dash number.
- b. Any special preservation, packaging, packing and marking required (see 5.0).

6.3 Definitions.

6.3.1 Lot size. A lot shall consist of all material manufactured in one continuous, unchanged production run.

6.4 Miscellaneous notes.

6.4.1 Order of precedence of this specification. This specification supersedes all others of the same scope. This specification was prepared for lack of a suitable document within the groupings of MIL-STD-143.

6.4.2 Approval procedure. When a supplier desires to obtain approval of his product in compliance with this specification, he should notify GD/Convair by letter of intent accompanied by three copies of a certified test report showing compliance. If after GD/Convair approval an alteration is made in the product, the approval is deemed cancelled unless GD/Convair has been notified of and approved the alteration.

6.4.3 Material sources.

6.4.3.1 Approved sources. The approved sources for the material described by this specification are as follows:

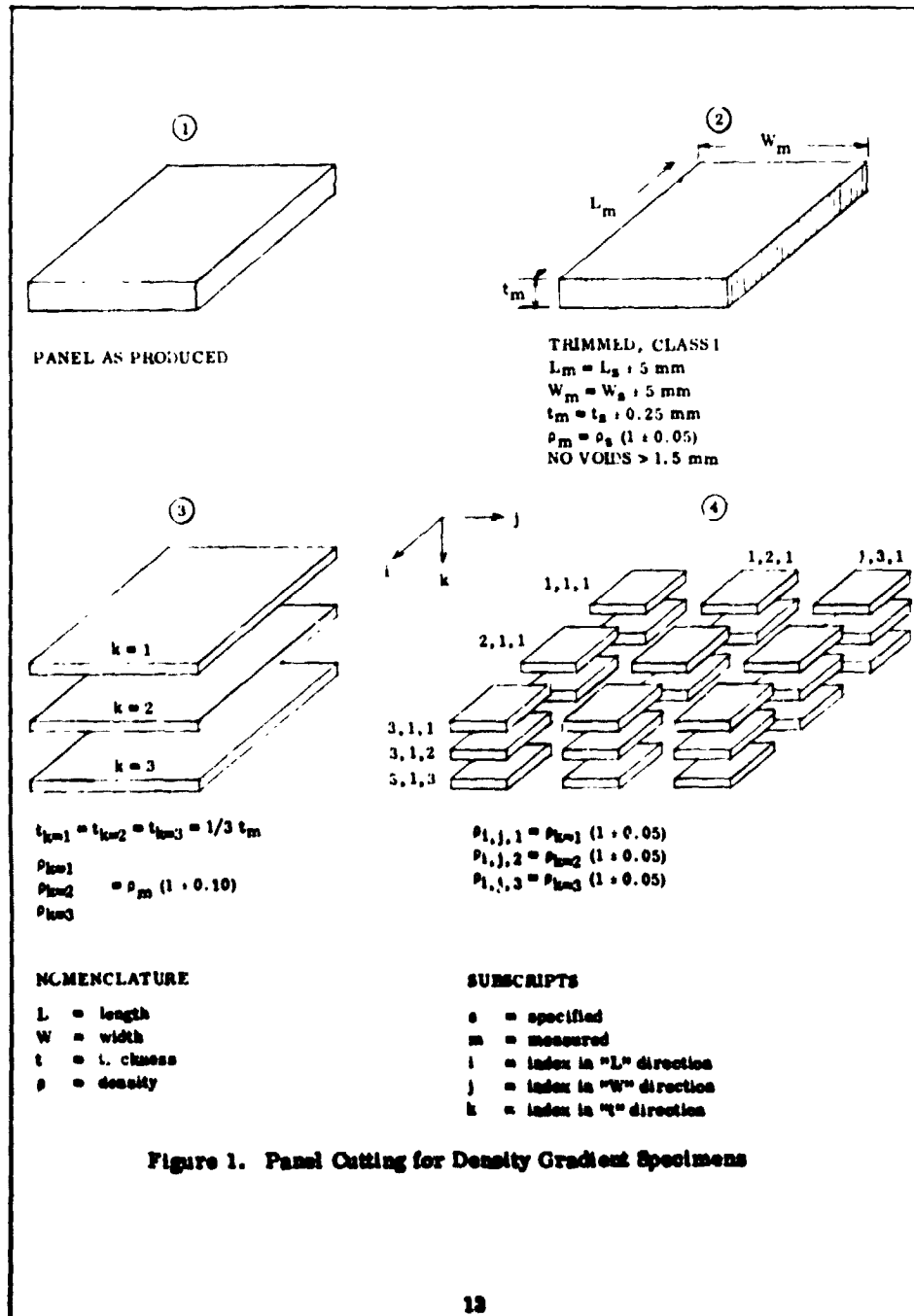
Plastics and Rubber Institute, TNO
97, Schoemakerstraat
P. O. Box 71
Delft, Holland

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6.4.4 Material identification numbers

GD/Convair Material Identification No.	Class Material	Nominal Density Kg/m ³	Thickness mm
0-06212 - 11	I	30	36
- 12	I	30	46
- 13	I	30	71
- 14	I	30	181
- 21	I	40	26
- 22	I	40	46
- 23	I	40	71
- 24	I	40	181
- 31	I	50	26
- 32	I	50	46
- 33	I	50	71
- 34	I	50	181
- 51	II	30	30
- 52	II	30	50
- 53	II	30	75
- 54	II	30	185
- 61	II	40	30
- 62	II	40	50
- 63	II	40	75
- 64	II	40	185
- 71	II	50	30
- 72	II	50	50
- 73	II	50	75
- 74	II	50	185



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Panel Number	Length		Width		Thickness Average mm	Bulk Density kg/m ³
	Average cm	Variation mm	Average cm	Variation mm		



Table 2. PPO Foam Panel Density Gradient Data Sheet

Panel No. _____

1. Specified Data for Trimmed Panel

$L_s =$ cm, $W_s =$ cm, $t_s =$ mm, $\rho_s =$ kg/m³

2. Trimmed Panel Measurements

	() _s - 5 mm	() _m	() _s + 5 mm	In Spec	Out of Spec
L					
W					
	() _s - 0.25 mm	() _m	() _s + 0.25 mm		
t					
	() _s × (1-0.05)	() _m	() _s × (1+0.05)		
ρ					

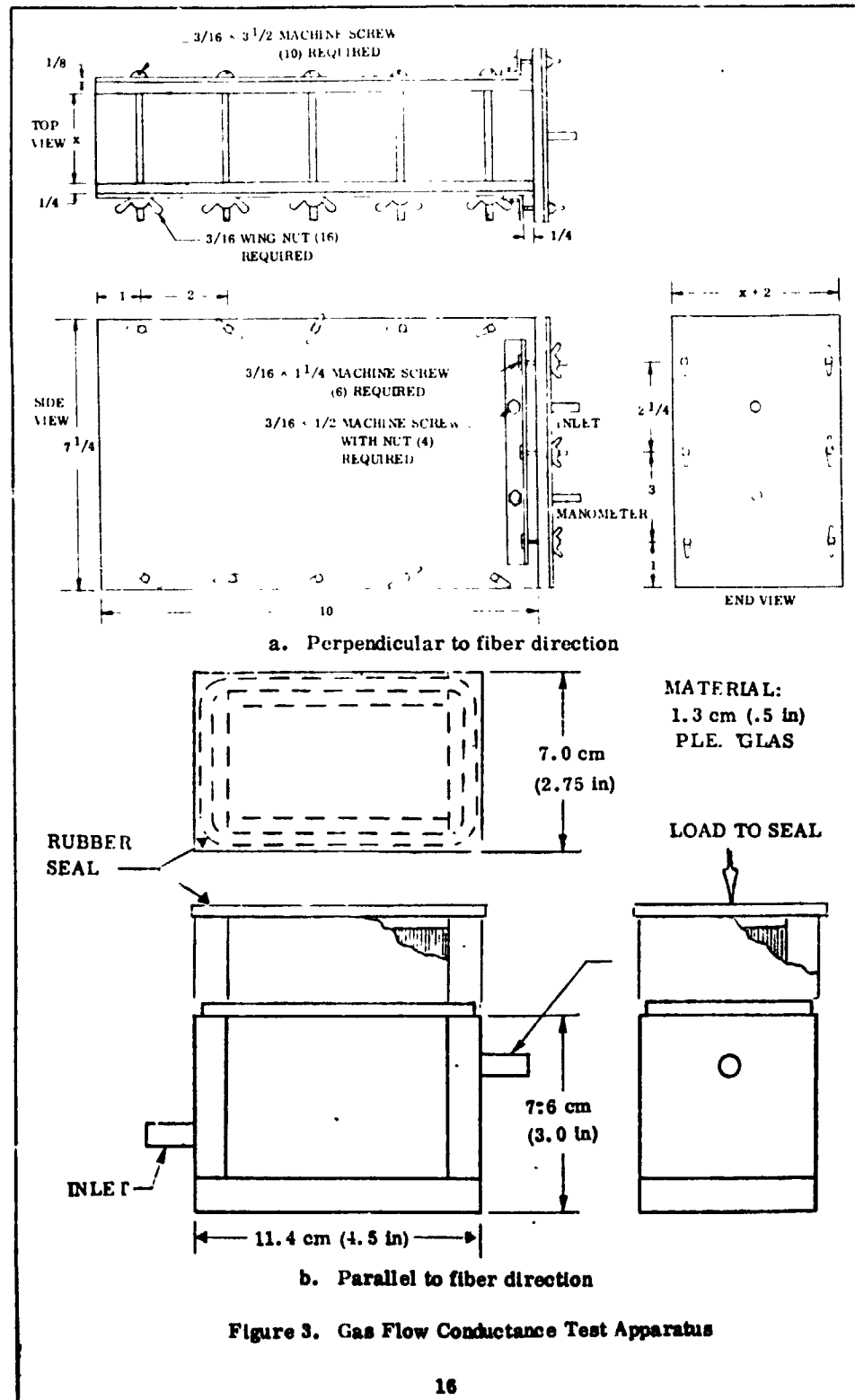
3. Sheet Measurements (Longitudinal Variation)

	$\rho_m \times (1-0.1)$	ρ_k	$\rho_m \times (1+0.1)$	In Spec	Out of Spec
ρ_m					
$\rho_{k=1}$					
$\rho_{k=2}$					
$\rho_{k=3}$					

4. Piece Measurements (Lateral Variation)

	k = 1		k = 2		k = 3	
	$\rho_k \times (1-0.05) \times (1+0.05)$		$\rho_k \times (1-0.05) \times (1+0.05)$		$\rho_k \times (1-0.05) \times (1+0.05)$	
ρ_k						
		In Out		In Out		In Out
$\rho_{1,1,k}$						
$\rho_{2,1,k}$						
$\rho_{3,1,k}$						
$\rho_{1,2,k}$						
$\rho_{2,2,k}$						
$\rho_{3,2,k}$						
$\rho_{1,3,k}$						
$\rho_{2,3,k}$						
$\rho_{3,3,k}$						

☐ Panel Accepted, ☐ Panel Rejected Reason:



Panel Number		Quadrant 2		Quadrant 3			
		Parallel Flow	Perpendicular Flow		Parallel Flow	Perpendicular Flow	
ΔP	\dot{w}_{pl}		\dot{w}_{pr}	$\frac{\dot{w}_{pr}}{\dot{w}_{pl}}$	ΔP	\dot{w}_{pl}	\dot{w}_{pr}
kN/m^2	$\text{cc}/(\text{sec} \cdot \text{m}^2)$				kN/m^2	$\text{cc}/(\text{sec} \cdot \text{m}^2)$	$\frac{\dot{w}_{pr}}{\dot{w}_{pl}}$
2.0					2.0		
4.0					4.0		
6.0					6.0		
8.0					8.0		
10.0					10.0		

Flow Area (m^2) = _____

= Thickness (mm) = _____

Panel Number _____ Shear			Tension			
Piece Number	Stress MN/m ²	Strain %	Modulus MN/m ²	Stress MN/m ²	Strain %	Modulus MN/m ²